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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

QUARTERLY PROGRESS REPORT NO. 21

For Quarter Ending July 15, 1970

prepared by
R. W. Harrison
J. P. Smith

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-6474
R. L. Davies and P. L. Stone, Project Managers
Materials and Structures Division

NUCLEAR SYSTEMS PROGRAMS
SPACE SYSTEMS
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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April 15, 1970 to July 15, 1970

August 12, 1970

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NASA Lewis Research Center
Cleveland, Ohio
R. L. Davies and P. L. Stone, Project Managers
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FOREWORD

The work described herein is sponsored by the National Aeronautics and Space Administration under Contract NAS 3-6474. R. L. Davies and P. L. Stone of NASA-Lewis Research Center are the NASA Technical Managers.

The program is being administered for the General Electric Company by E. E. Hoffman, and R. W. Harrison is acting as the Program Manager. Personnel making major contributions to the program during the current reporting period include:

T-111 Corrosion Loop Posttest Evaluation - J. Smith, A. Losekamp

Chemical Analysis - H. Bradley, L. Paian

1900°F Lithium Loop - J. Smith, T. Irwin

Advanced Tantalum Alloy Capsule Tests - G. Brandenburg

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from April 15, 1970 to July 15, 1970 of a three part program.

A. T-111 Rankine System Corrosion Test Loop

The primary task of this program is to fabricate, operate for 10,000 hours, and evaluate a T-111 Rankine System Corrosion Test Loop. Materials for evaluation include the containment alloy, T-111 (Ta-8W-2Hf) and the turbine candidate materials Mo-TZC and Cb-132M which are located in the turbine simulator of the two-phase potassium circuit of the system. The loop design is similar to the Cb-1Zr Rankine System Corrosion Test Loop; a two-phase, forced convection, potassium corrosion test loop which was tested under Contract NAS 3-2547.[1] Lithium was heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary potassium loop was accomplished by radiation in a high vacuum environment to the water cooled chamber. The compatibility of the selected materials is being evaluated at conditions representative of space electric power system operating conditions, namely:

a. Boiling temperature, 2050°F
b. Superheat temperature, 2150°F
c. Condensing temperature, 1400°F
d. Subcooling temperature, 1000°F
e. Mass flow rate, 40 lb/hr
f. Boiler exit vapor velocity, 50 ft/sec
g. Average heat flux in plug (0-18 inches), 240,000 Btu/hr ft^2
h. Average heat flux in boiler (0-250 inches), 23,000 Btu/hr ft^2

This loop completed 10,000 hours of testing in March 1970 and is undergoing evaluation.

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(1) Hoffman, E. E. and Holowach, J., Cb-1Zr Rankine System Corrosion Test Loop, Potassium Corrosion Test Loop Development Topical Report No. 7, NASA-CR-1509, 1970.

B. 1900°F Lithium Loop

Also included in the program is the fabrication, 7500-hour operation, and evaluation of a 1900°F, high flow velocity (1 gpm), pumped lithium loop designed to evaluate the compatibility of T-111 clad UN fuel specimens, ASTAR 811 type alloys, T-111, Mo-TZM and W-Re-Mo Alloy 256,* at conditions simulating a space power reactor system. The planned schedule for this loop is a 2500 hour test with replacement of two fuel capsules and then an additional 5000 hour test of the loop.

C. Advanced Tantalum Alloy Capsule Tests

The program also included capsule testing to evaluate advanced tantalum alloys of the ASTAR 811 type (Ta-8W-1Re-1Hf) in both potassium and lithium. Refluxing potassium capsule tests at 2200°F and lithium thermal convection capsule tests at 2400°F completed 5000 hours of testing, and the test specimens were evaluated. The results of these tests have been reported in previous quarterly reports; therefore, they are not discussed in this report.

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*W-25 a/o Re-30 a/o Mo (W-29 w/o Re-18 w/o Mo)

II. SUMMARY

Posttest evaluation of the loop components from the 10,000 hours testing of the T-111 Rankine System Corrosion Test Loop is well underway. Most of the chemical analyses are complete and metallographic examination has been initiated. Initial chemical analyses indicate some depletion of oxygen from the T-111 by the lithium. Evidence of higher oxygen in the potassium circuit has been observed; however, more analyses are needed to verify the oxygen distribution.

The 1900°F Lithium Loop successfully completed the initial 2500 hours of testing. As planned, the loop was shut-down after this initial test period, and the fuel test section and associated specimens were removed for examination. New specimens have been added, and the fuel test section has been successfully re-welded into the loop. The loop is in the final stages of being restarted for the remaining 5000 hours of testing. Initial results of the fuel specimen examination indicate no deleterious effects of the 2500 hour lithium exposure.

III. PROGRAM STATUS

A. T-111 Rankine System Corrosion Test Loop

1. Removal of Residual Alkali Metal

After completion of visual inspection, the boiler-turbine simulator assembly was transferred to the weld chamber. The chamber was evacuated and back-filled with argon, and valves were attached to the lithium and the potassium circuits to facilitate removal of residual alkali metals with liquid ammonia. The assembly was removed from the weld chamber and connected to the liquid ammonia system described previously.[1] The assembly was maintained at -50°C in a liquid nitrogen cooled, 50 percent (by volume) mixture of methanol and water held in insulated containers as shown in Figure 1. When the assembly reached -50°C, the temperature necessary to maintain the ammonia in a liquid state, the argon flow valve was closed and the valve to the ammonia bottle opened. The ammonia flow through the assembly was maintained until the effluent was clear and colorless. The lithium circuit contained a greater quantity of alkali metal than the potassium circuit and required a greater quantity of liquid ammonia to remove the lithium. Similar observations were made when the alkali metals were removed from the boiler previously.[1] Each circuit of the assembly was flushed with distilled water and ethyl alcohol and allowed to dry.

2. Final Preparation for Posttest Evaluation

To obtain all of the desired chemistry, metallographic and other type of specimens needed for posttest evaluation, the boiler-turbine assembly was cut into the five following major subassemblies:

1) Plug section of boiler
2) Boiler
3) First stage turbine simulator
4) Potassium vapor crossover line

[1] Advanced Refractory Alloy Corrosion Loop Program Quarterly Progress Report #13, Period Ending July 15, 1968, NASA-CR-72483, pp 27.

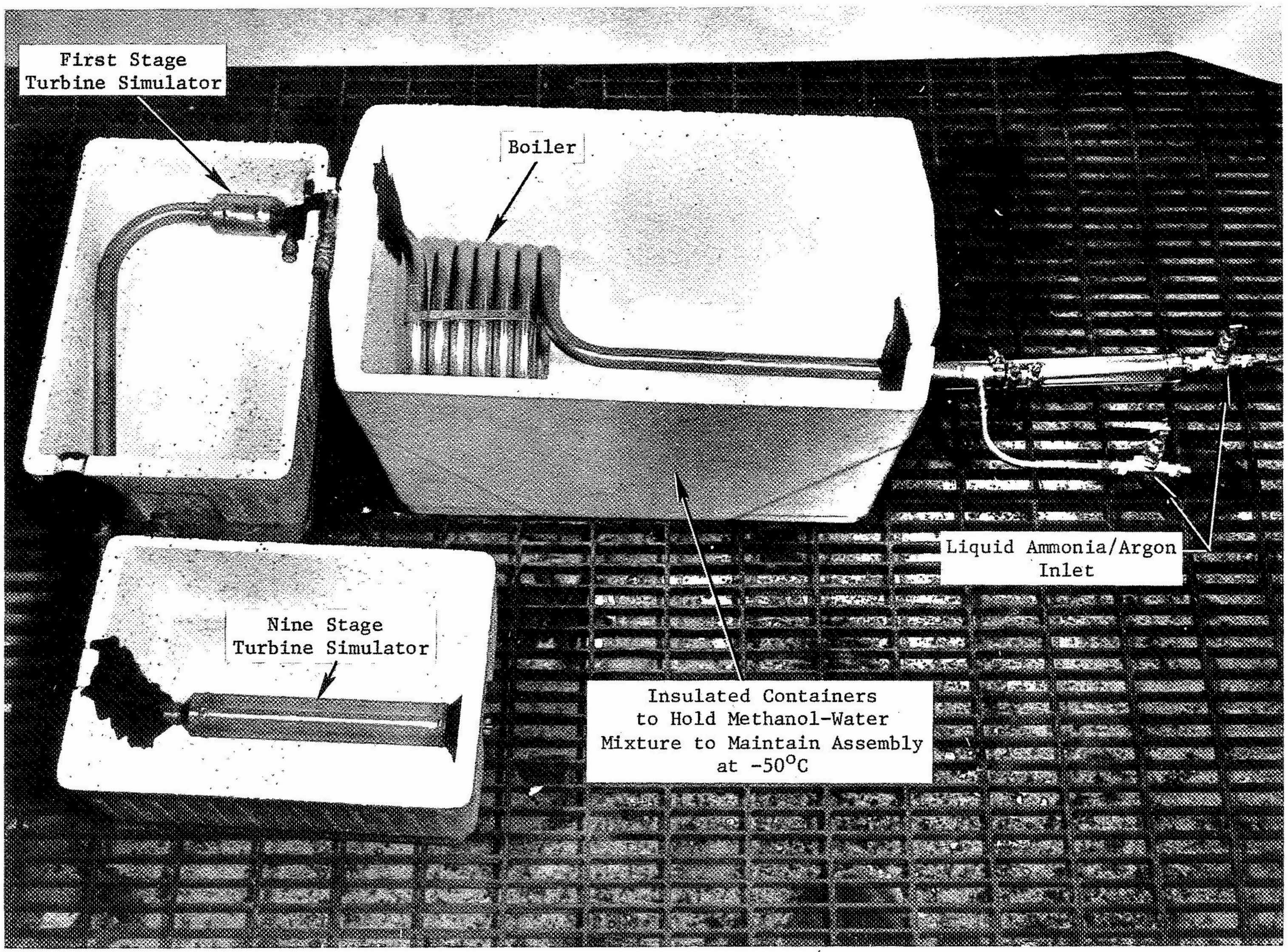

Figure 1. Set-Up for the Removal of Residual Alkali Metals from T-111 Rankine System Corrosion Test Loop Components by Reaction with Liquid Ammonia. (P70-4-18B)

5) Nine stage turbine simulator

These subassemblies were further dissected, as described below, to facilitate further visual examination and obtain necessary specimens.

a) Plug section of boiler

The plug section of the boiler was disassembled as shown in Figure 2 by filing the welds at the bottom fitting and at the bottom of the swirler wire. The most interesting observations were made on the 3/8-inch potassium containment tube which is shown in Figure 3. Of particular interest is the gold colored deposit on the OD of the tube. The gold colored deposit, which has been identified as HfN by x-ray diffraction techniques, appeared heaviest in the region of the fitting shown in the inset of Figure 3. The transition from the lower tube area containing the HfN deposit to the upper tube area without deposit is believed to be the area at which the ID of the 3/8-inch tube wall becomes exposed to essentially dry potassium vapor; whereas, the liquid is confined to the centerbody-swirler wire insert. The helical shape of the transition probably results from the potassium flow pattern induced by the swirler wire. No deposit or discoloration was observed on the swirler wire-plug insert, as can be seen in Figure 3.

b) Boiler

The boiler was further cut as shown in Figure 4 to allow closer examination of each coil. Initial plans were to remove the 3/8-inch tube from each coil; however, the eliptical shape of the 1-inch lithium containment tube resulting from the forming operation prevented removal of the spacers holding the 3/8-inch tube in place. Because of this problem only two coils were cut-up for complete examination. The coil of primary interest contained the boiler repair welds[2] and is shown in Figure 5. As can be seen in the inset, no evidence of corrosion or discoloration was observed.

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[2] Advanced Refractory Alloy Corrosion Loop Program Quarterly Progress Report #14, Period Ending October 1968, NASA-CR-72505 (GESP-189) p 14.

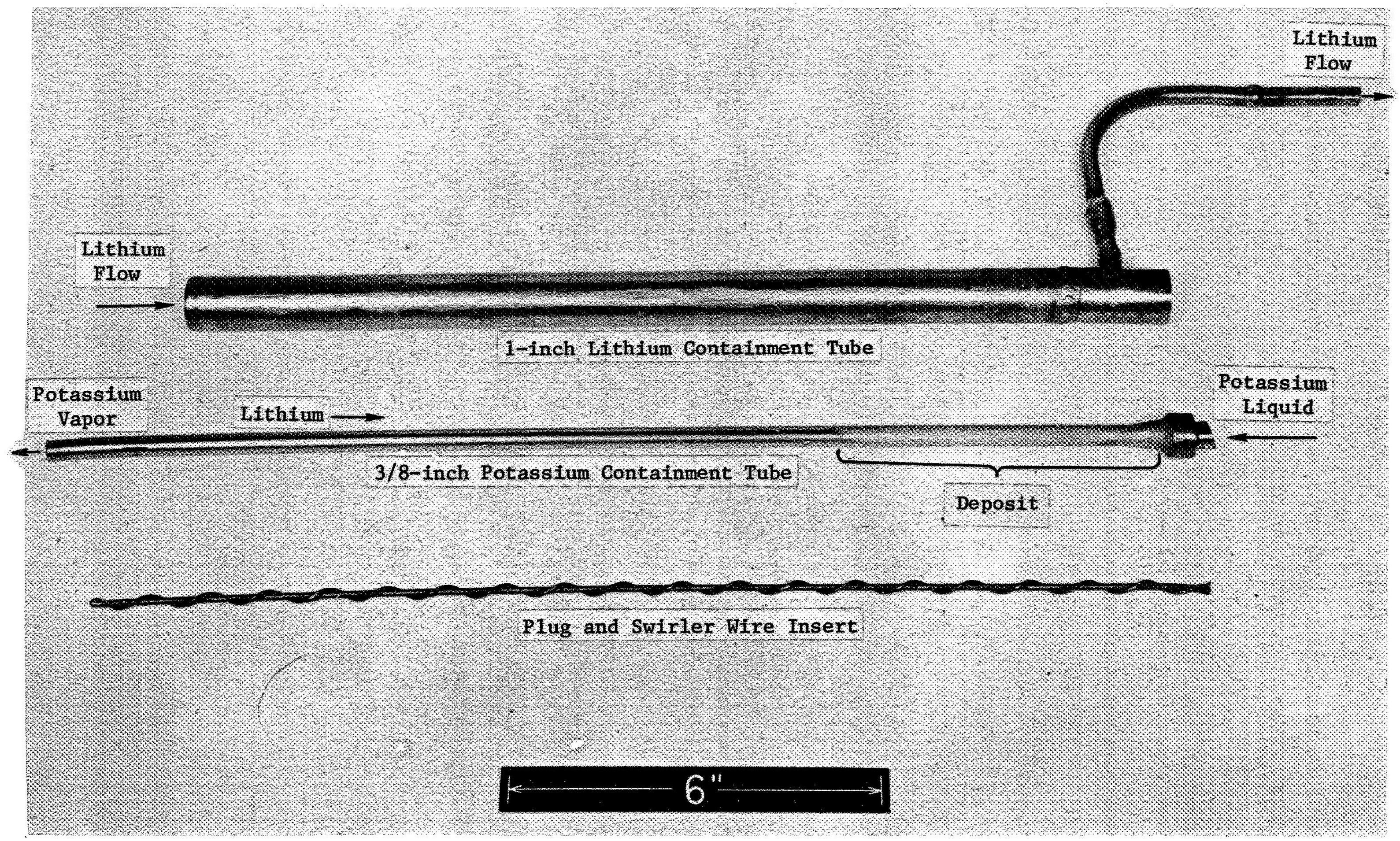

Figure 2. Components of Boiler Plug Section of T-111 Rankine System Corrosion Test Loop Following 10,000 Hours of Continuous Operation. (Orig. P70-5-1M)

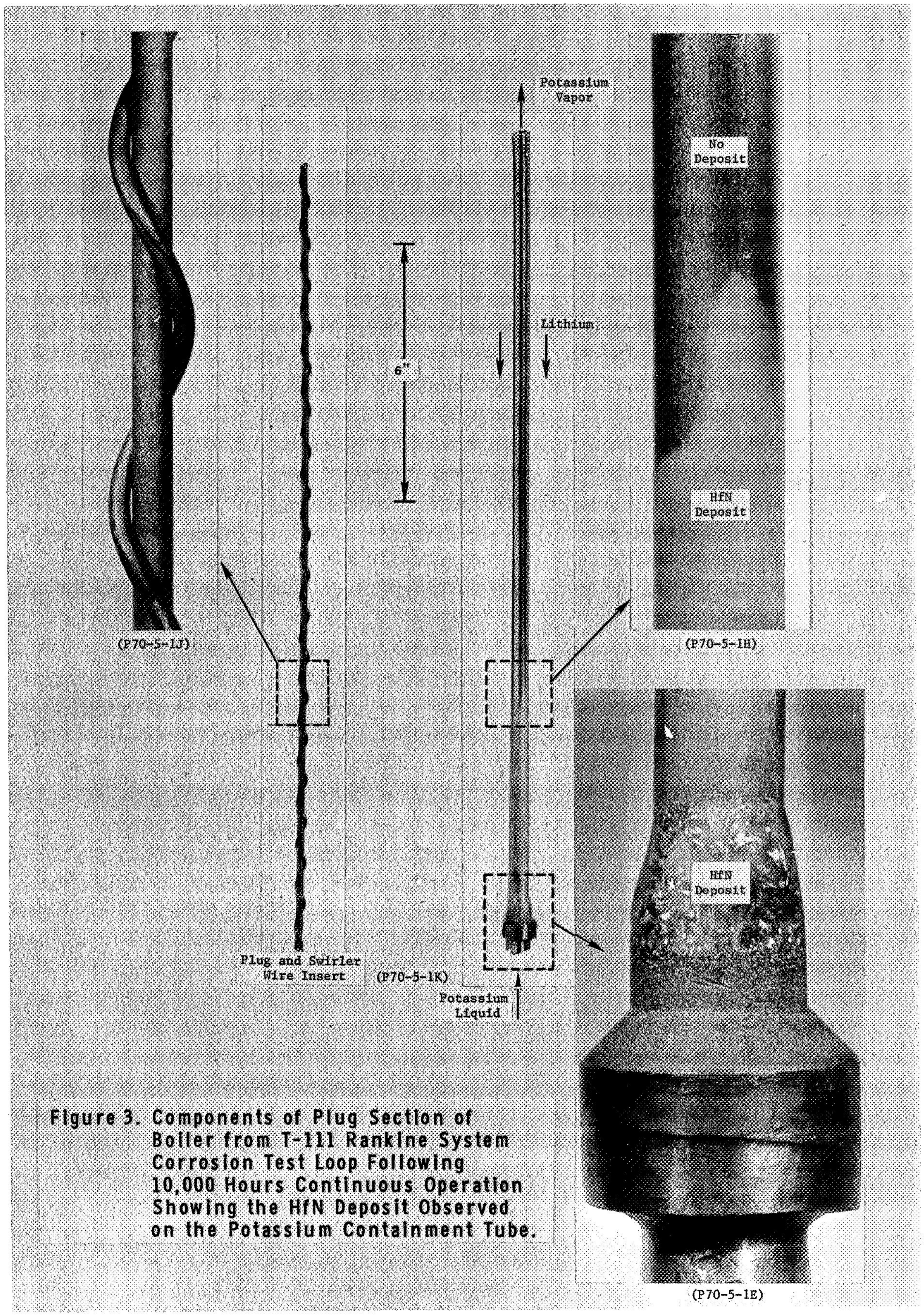

Figure 3. Components of Plug Section of Boiler from T-111 Rankine System Corrosion Test Loop Following 10,000 Hours Continuous Operation Showing the HfN Deposit Observed on the Potassium Containment Tube.

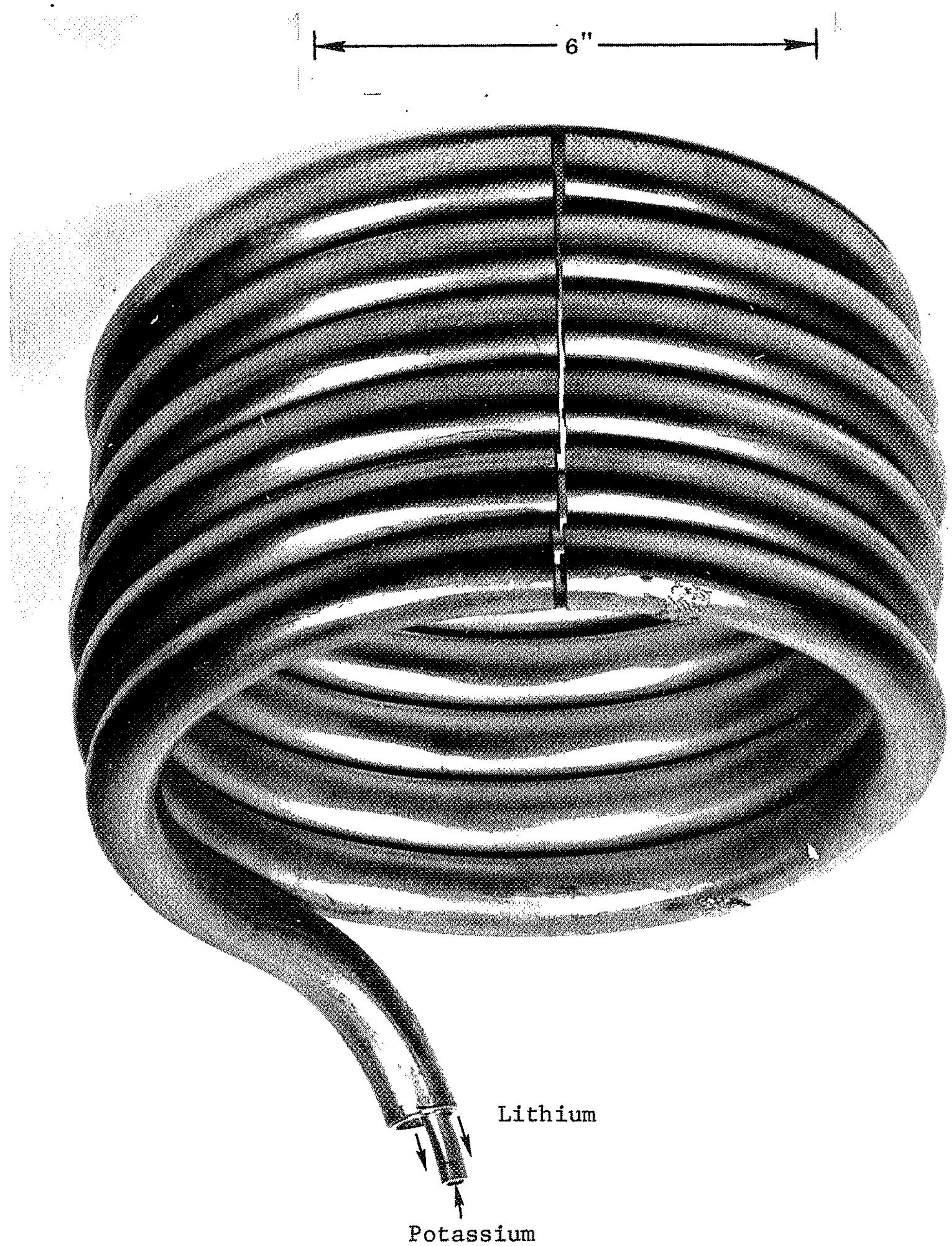

Figure 4. Boiler from T-111 Rankine System Corrosion Test Loop Following 10,000 Hours of Continuous Operation Illustrating Location of Cut Providing Removal and Inspection of Individual Coils. (Orig. P70-5-12A)

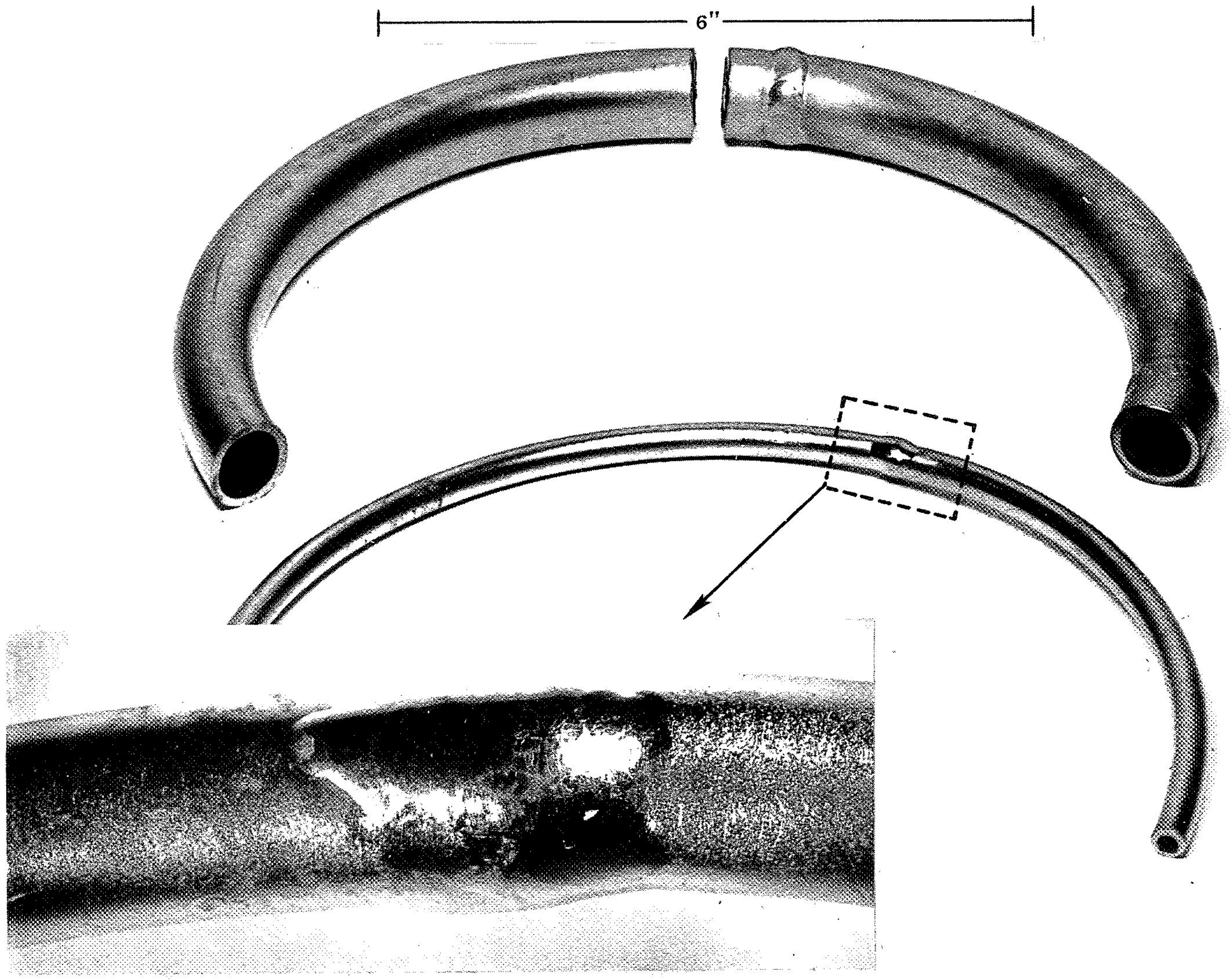

Figure 5. Repair Welds in Boiler Tubes of T-111 Rankine System Corrosion Test Loop Following 10,000 Hours of Continuous Operation. (Orig. P70-5-12E, P70-5-12B)

c) Turbine simulators

The nozzles and blades were removed from the turbine simulators by making a longitudinal cut in each of the housings, as described previously.[3] The appearance of all blades and nozzles was very good, and no signs of gross corrosion or erosion were observed. Potassium vapor flow patterns were observed on the blades, as seen in Figures 6 and 7. The flow patterns appear merely as very thin areas of discoloration on the impingement surface and do not represent any serious build-up or deterioration. As can be seen in Figure 6, the Stage 1 nozzle still has a part of the T-111 housing attached to it; this could not be removed because the nozzle had become securely bonded to the end plug during the 10,000 hour exposure apparently due to the very tight fit of the mating components. The nozzles of the first, second, sixth and tenth stages were sectioned longitudinally to allow for better visual examination. As can be seen in Figure 8, no deterioration of the nozzles occurred.

3. Posttest Evaluation

a) Metallographic examination

Metallographic examination will be performed on specimens cut from the locations shown in Figure 9. Specimens from both the lithium and potassium circuits will be examined and the microstructures compared with untested (pretest) specimens from the same lot of material. All pretest specimens were heat treated for 1 hour at 2400°F to simulate the loop postweld anneal. Differences in microstructure observed would therefore be a result of the test exposure. Special emphasis will be placed on the examination of the welds made during the repair and replacement of the boiler for any signs of corrosion since this loop was the first of its kind to be repaired and subsequently tested successfully without difficulty.

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(3) Hoffman, E. E. and Holowach, J., Cb-1Zr Rankine System Corrosion Test Loop, Potassium Corrosion Test Loop Development Topical Report No. 7, NASA-CR-1509 (GESP-246) pp 236.

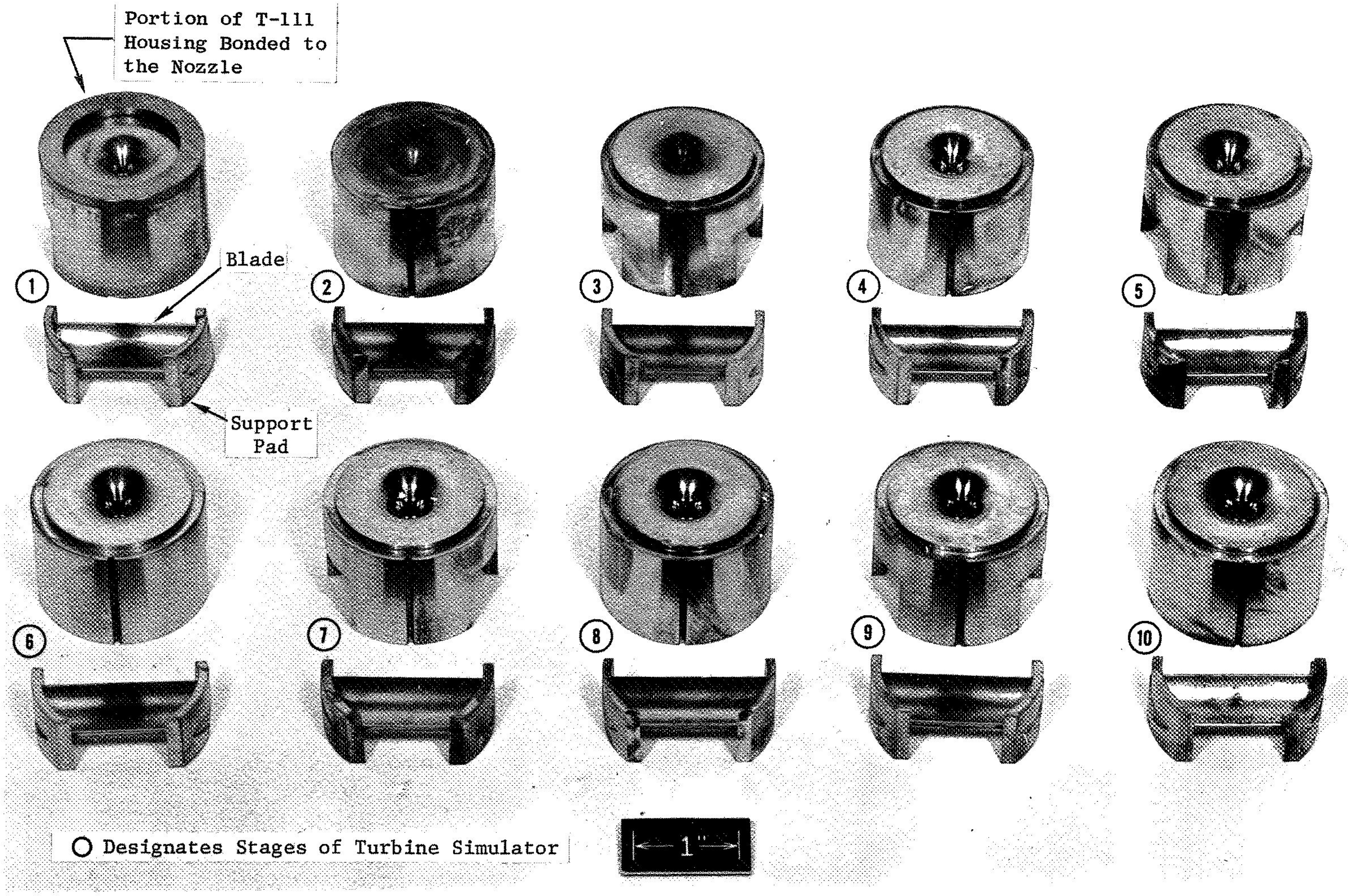

Figure 6. Turbine Simulator Nozzles, Blades and Support Pads Following 10,000 Hours of Continuous Exposure to Potassium Vapor in the T-111 Rankine System Corrosion Test Loop. All are Mo-TZC Except Stages 6 and 9 Which are Cb-132M. (Orig. P70-5-6M)

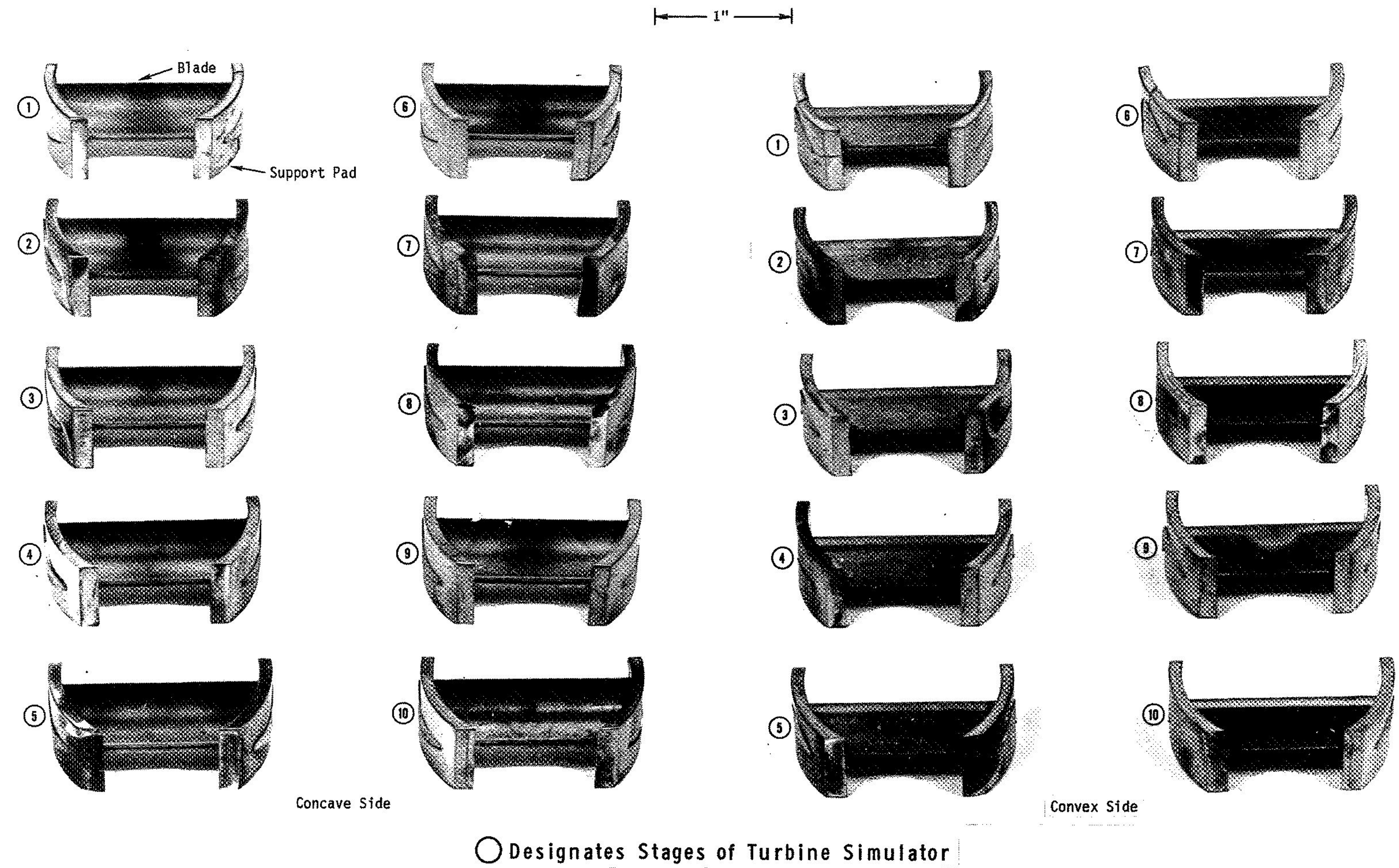

Figure 7. Concave and Convex Side of Turbine Simulator Blades Following 10,000 Hours Continuous Exposure to Potassium Vapor in the T-111 Rankine System Corrosion Test Loop. All are Mo-TZC Except Stages 6 and 9 Which are Cb-132M. (Orig. P70-5-6P and P70-5-6N)

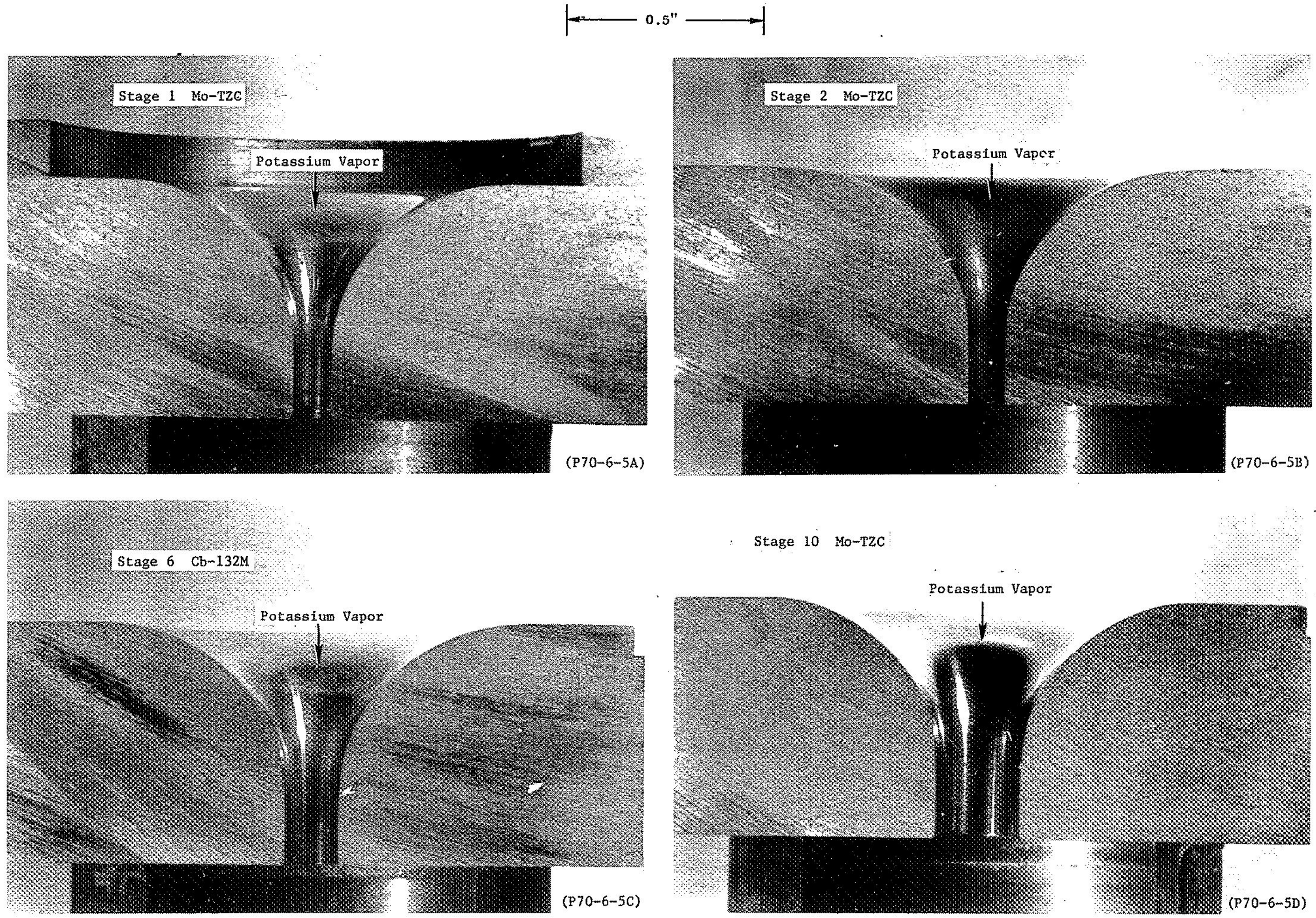

Figure 8. Longitudinal Cross Section of Turbine Simulator Nozzles Following 10,000 Hours of Continuous Exposure to Potassium Vapor in the T-111 Rankine System Corrosion Test Loop. (P70-6-5A) (P70-6-5B) (P70-6-5C) (P70-6-5D)

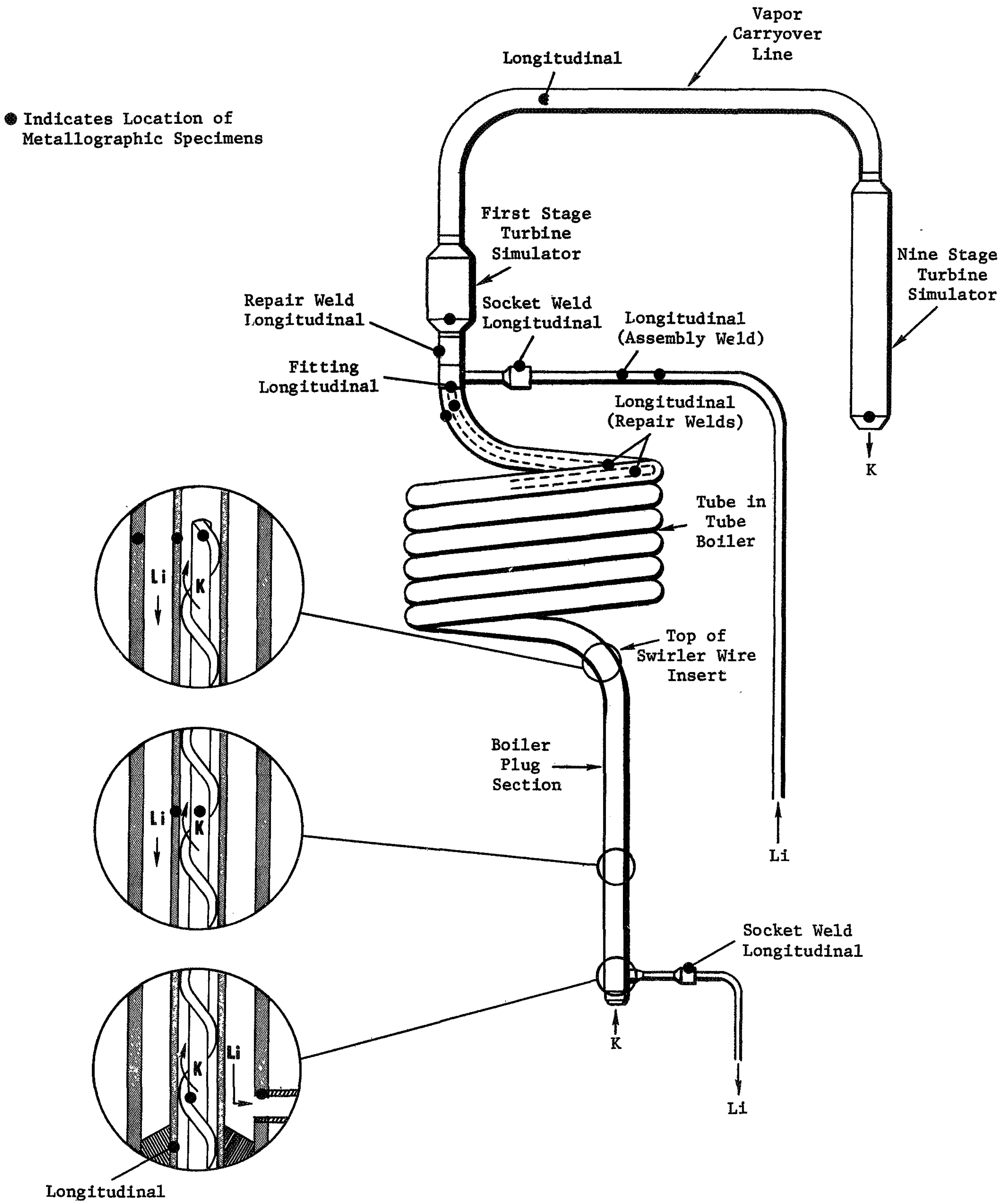

Figure 9. Location of Specimens for Metallographic Examination from the T-111 Rankine System Corrosion Test Loop Following 10,000 Hours of Continuous Operation. (All are Transverse Sections Unless Noted Otherwise.)

All metallography specimens have been nickel plated (for edge preservation), mounted, polished and are ready for final examination.

b) Chemical analysis

Chemical analyses for C, O, H, N have been obtained on specimens cut from the locations shown in Figure 10. The results of all analyses from loop components performed to date are presented in Tables I, II and III. The results of oxygen analysis are summarized in Figure 11.

The oxygen concentrations in the 1-inch diameter T-111 tubing, the ID of which was exposed to lithium, are all ≤ 10 ppm. The dissolution of oxygen from the T-111 by the lithium is clearly indicated and has been observed previously.[4] The oxygen concentrations in the 3/8-inch diameter T-111 tubing exposed to lithium on the OD and potassium on the ID are less consistent, with some large oxygen concentrations indicated. The analytical results of the inner and outer wall segment specimens coupled with the oxygen dissolution observed in the 1-inch diameter tube indicates the high oxygen (> 50 ppm) source is the potassium. Two high oxygen peaks were observed; the first was found 5 to 6 inches from the lithium exit (boiler inlet), and the second was found 16 to 18 inches from the lithium exit. It is currently believed that the first oxygen peak is associated with the region where the inner wall of the potassium containment tube (3/8-inch) becomes essentially dry. From this region to the end of the plug section most of the remaining potassium liquid is carried along on the swirler wire and centerbody. It is hypothesized that as liquid potassium is converted to vapor in this area, the oxygen concentration of the remaining liquid becomes increasingly higher until it finally is concentrated at the wall and contaminates the T-111 either by absorption and interstitial diffusion and/or by reaction, possibly corrosion. Metallographic examination of specimens from this area should elucidate this postulate.

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[4] Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 18 for Period Ending October 15, 1969, NASA Contract NAS 3-6474, NASA-CR-72620, p. 32.

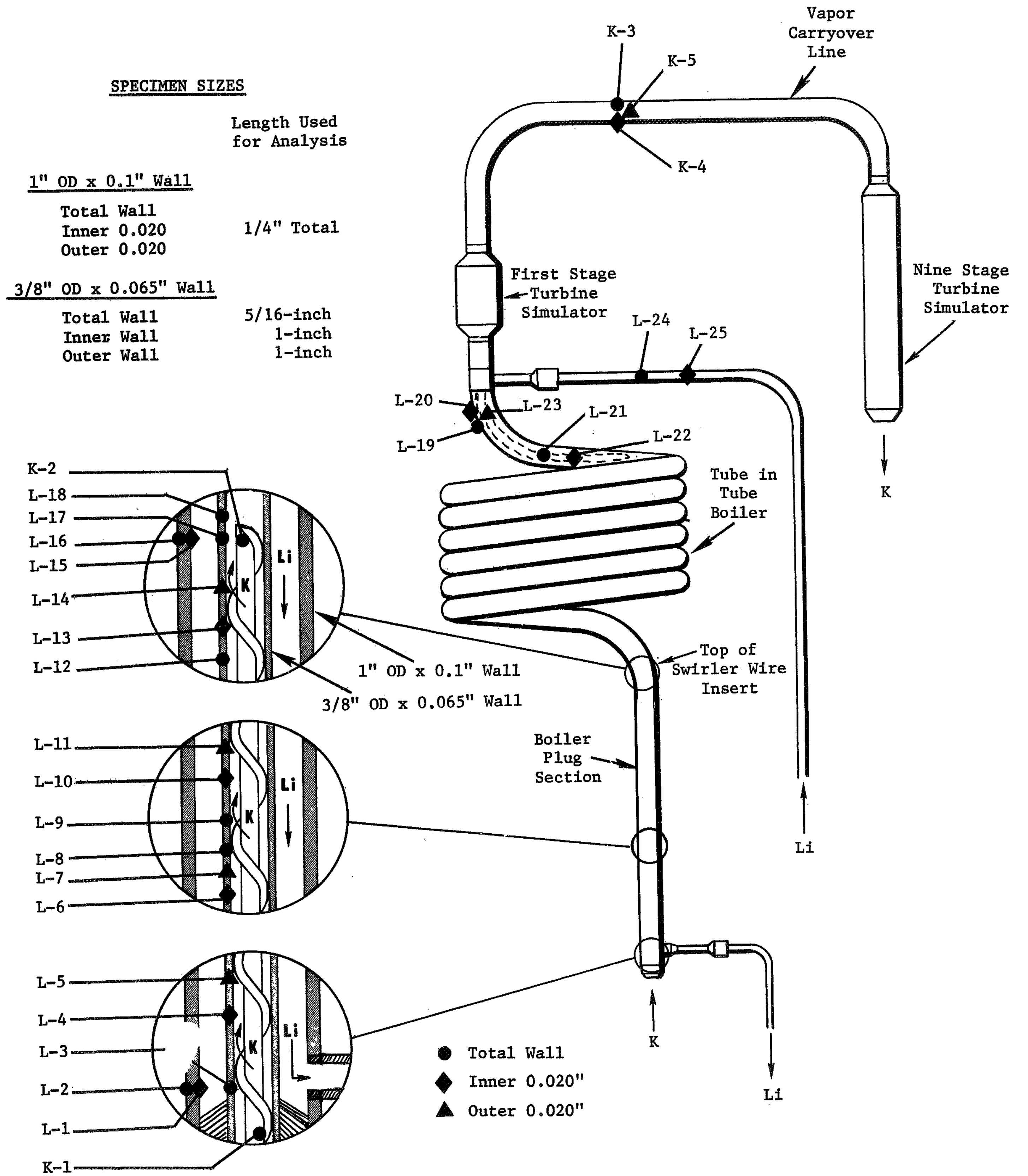

Figure 10. Location of Specimens Used for Chemical Analyses of Loop Components from the T-111 Rankine System Corrosion Test Loop Following 10,000 Hours of Continuous Operation.

TABLE I. RESULTS OF CHEMICAL ANALYSIS OF SPECIMENS OF T-111 ALLOY LITHIUM CONTAINMENT TUBE[a] FROM THE BOILER

Specimen Identification and Description	Temperature °F		Chemical Analysis, ppm O	N	H	C
(L-1)[b] Bottom of plug section at lithium exit, inner 0.020 inch of wall	2128		1	18	< 1	19
			2	19	1	17
		Avg.	1	18	< 1	18
(L-2) Bottom of plug section at lithium exit, total wall	2128		11	14	< 1	16
			10	13		16
		Avg.	10	13	< 1	16
(L-15) Top of plug section 18 inches from lithium exit, inner 0.020 inch of wall	2200		8	16	< 1	25
			12	21		24
		Avg.	10	18	< 1	24
(L-16) Top of plug section 18 inches from lithium exit, total wall	2200		2	13	< 1	34
			2	14		34
		Avg.	2	13	< 1	34
(L-19) Top of boiler 1 1/2 inches from lithium inlet, total wall	2244		5	13	< 1	37
			6	12		49
		Avg.	5	12	< 1	43
(L-20) Top of boiler 1 1/2 inches from lithium inlet, inner 0.020 inch of wall	2244		1	14	< 1	37
			4	15		46
		Avg.	2	14	< 1	41
Before test			17	2	1	44

a) 1-inch OD x 0.10-inch wall thickness.

b) Specimen location indicated in Figure 10.

TABLE II. RESULTS OF CHEMICAL ANALYSIS OF SPECIMENS OF T-111 ALLOY POTASSIUM CONTAINMENT TUBE[a] FROM THE BOILER

Specimen Identification and Description		Estimated Temperature °F		Chemical Analysis, ppm O	N	H	C
(L-3)	Bottom of plug section at lithium exit, total wall	1900		2	73	1	32
				7	75	1	34
			Avg.	4	74	1	34
(L-4)	Bottom of plug section, 1/2 inch from lithium exit, inner 0.020 inch of wall	1925		14	15	< 1	54
				16	20		61
			Avg.	15	17	< 1	57
(L-5)	Bottom of plug section, 1 1/2 inch from lithium exit, outer 0.020 inch of wall	1950		30	217	2	91
				37	90	1	94
			Avg.	33	153	1	92
(L-6)	2 1/2 inches from lithium exit, inner 0.020 inch of wall	2050		14	26	1	66
				18	27	1	45
			Avg.	16	26	1	55
(L-7)	3 1/2 inches from lithium exit, outer 0.020 inch of wall	2050		25	67	1	62
				27	38	1	81
			Avg.	26	52	1	71
(L-8)	4 inches from lithium exit, total wall	2050		6	41	2	31
				10	65	1	43
			Avg.	8	53	1	37
(L-9)	5 inches from lithium exit, total wall	2040		378	26	5	36
				236	23	3	41
			Avg.	307	24	4	38
(L-10)	5 1/2 inches from lithium exit, inner 0.020 inch of wall	2040		1051	9	10	40
				845	12	5	49
			Avg.	948	10	7	44
(L-11)	6 1/2 inches from lithium exit, outer 0.020 inch of wall	2040		4	16	< 1	46
				8	21	1	42
			Avg.	6	18	< 1	44
(L-12)	Top of plug section 14 inches from lithium exit, total wall	2040		56	17	1	
				32	17	1	
			Avg.	44	17	1	
(L-13)	Top of plug section 14 3/4 inches from lithium exit, inner 0.020 inch of wall	2040		7	14	1	55
				3	20	< 1	52
			Avg.	5	17	< 1	53
(L-14)	Top of plug section 15 3/4 inches from lithium exit, outer 0.020 inch of wall	2040		4	11	< 1	47
				2	11	2	36
			Avg.	3	11	1	41
(L-17)	Top of plug section 16 3/4 inches from lithium exit, total wall	2040		545	30	4	47
				648	28	10	39
			Avg.	596	29	7	43
(L-18)	Top of plug section 17 1/4 inches from lithium exit, total wall	2040		789	17	5	
				1414	16	5	
			Avg.	1101	16	5	
(L-21)*	Top of boiler 6 1/2 inches from lithium inlet, total wall	2140		3	9	< 1	24
				2	8		25
			Avg.	2	8	< 1	24
(L-22)*	Top of boiler 6 1/2 inches from lithium inlet, inner 0.020 inch of wall	2140		4	3	2	63
				1	3	1	63
			Avg.	2	3	1	63
(L-23)*	Top of boiler 1 1/2 inches from lithium inlet, outer 0.020 inch of wall	2140		2	10	2	43
				4	9	1	33
			Avg.	3	9	1	38
	Before test, original boiler tube			16	5	1	42
	*Before test, replacement tube used for boiler repair			8	2	< 1	29

a) 3/8-inch OD x 0.065-inch wall thickness.

TABLE III. RESULTS OF CHEMICAL ANALYSIS OF MISCELLANEOUS SPECIMENS OF T-111 ALLOY EXPOSED TO POTASSIUM OR LITHIUM DURING THE 10,000-HOUR TEST

	Specimen Identification and Description	Temperature °F		Chemical Analysis, ppm O	N	H	C
	Potassium Circuit						
(K-1)	Center body and swirl wire,[a] bottom of plug section	1850		53	8	2	51
				36	7	1	48
			Avg.	44	7	1	49
(K-2)	Center body and swirl wire,[a] top of plug section	2040		196	7	3	31
				271	5	2	45
			Avg.	233	6	2	38
	Before test			8	3	8	24
(K-3)	Vapor carryover tube,[b] uninsulated, 1/2 inch from end of insulation, total wall	1900[c]		38	7	1	71
				24	5	< 1	61
			Avg.	31	6	< 1	66
(K-4)	Vapor carryover tube, uninsulated, 1/2 inch from end of insulation, inner 0.020 inch of wall	1900[c]		17	4	< 1	67
				16	4		67
			Avg.	16	4	< 1	67
(K-5)	Vapor carryover tube, uninsulated, 1/2 inch from end of insulation, outer 0.020 inch of wall	1900[c]		50	4	1	58
				53	6	1	67
			Avg.	51	5	1	62
	Before test			17	2	1	44
	Lithium Circuit						
(L-24)	Lithium heater exit tube,[d] 9 inches from boiler inlet, total wall	2250		4	4	< 1	23
				6	1		30
			Avg.	5	2	< 1	26
(L-25)	Lithium heater exit tube,[d] 9 inches from boiler inlet, inner 0.020 inch of wall	2250		11	3	< 1	39
				17	10		50
			Avg.	14	6	< 1	44
	Before test			16	5	1	42

a) 0.060-inch swirl wire wrapped on and tack welded to 0.125-inch-OD center body.
b) 1-inch OD x 0.10-inch wall thickness.
c) Estimated temperature.
d) 3/8-inch OD x 0.065-inch wall thickness.

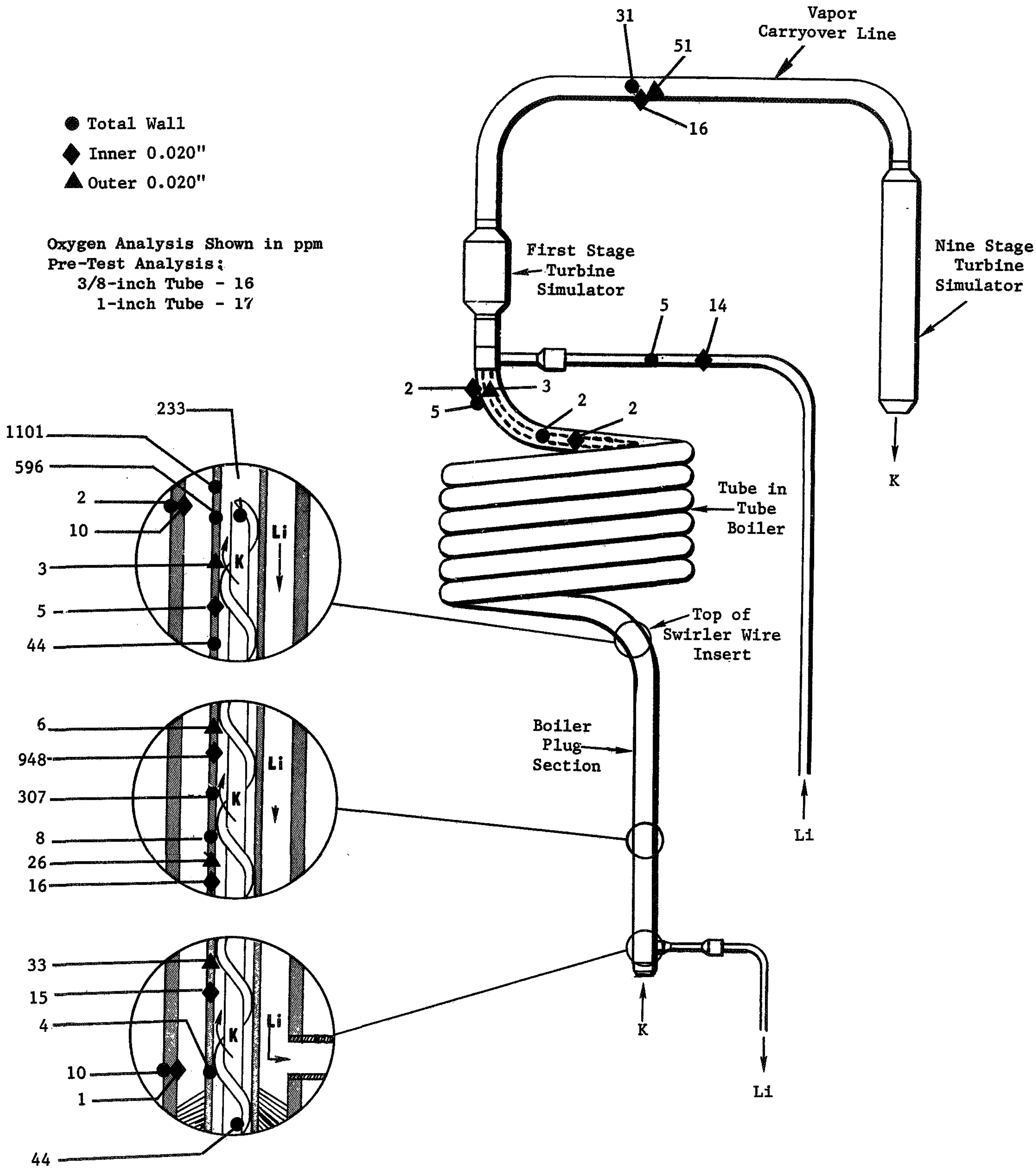

Figure 11. Results of Oxygen Analysis of Loop Components from the T-111 Rankine System Corrosion Test Loop Following 10,000 Hours of Continuous Operation.

The area of the second oxygen peak corresponds to the end of the swirler wire-plug insert. A recently completed experimental study at GE-NSP on air-water flow in tubes containing swirl generators[5] had indicated the behavior of liquid in tubes containing plug inserts similar to that employed in the T-111 Corrosion Loop. The results of the air-water-flow study[5] as applied to the T-111 Corrosion Loop would indicate that the ID of the potassium containment tube will become void of liquid potassium; however, liquid potassium will still flow up the plug insert since the pressure is low in the center. At the end of the insert, this liquid potassium is thrown to the ID of the potassium containment tube where it immediately is converted to vapor. The transfer of liquid from the plug insert to the containment tube wall is illustrated for the air-water flow tests in Figure 12.[5] Since the air water tests are performed at ambient temperatures; the conversion to vapor on contact with the tube wall does not occur as it does for the case when the tube walls are hot such, as in a boiler. Oxygen contamination of the T-111 wall in this area can therefore occur by the same process as previously described. The oxygen concentrations of the centerbody and swirl wire at the top end of the plug were also high, supporting this postulate.

A comparison of the oxygen concentration in the inner and outer wall segments of the uninsulated vapor-carryover line indicates very minor oxygen pickup (30 ppm) in the T-111 from the chamber environment during the 10,000 hour test.

As shown by item L-5 in Table II, the nitrogen and carbon concentrations in the outer wall specimens taken from the 3/8-inch potassium containment tube in the area of the HfN deposit are higher than for any other evaluated portion of the loop. Since essentially all of the carbon and nitrogen concentrations shown in Table I, II and III are at least equal to the pretest value, it must be assumed that the source of these elements is the unevaluated portions of the lithium circuit.

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(5) Bond, J. A., The Design of Components for an Advanced Rankine Cycle Test Facility, presented at: Fifth Intersociety Energy Conversion Engineering Conference in Las Vegas, Nevada, September 21-25, 1970, Figure 8.

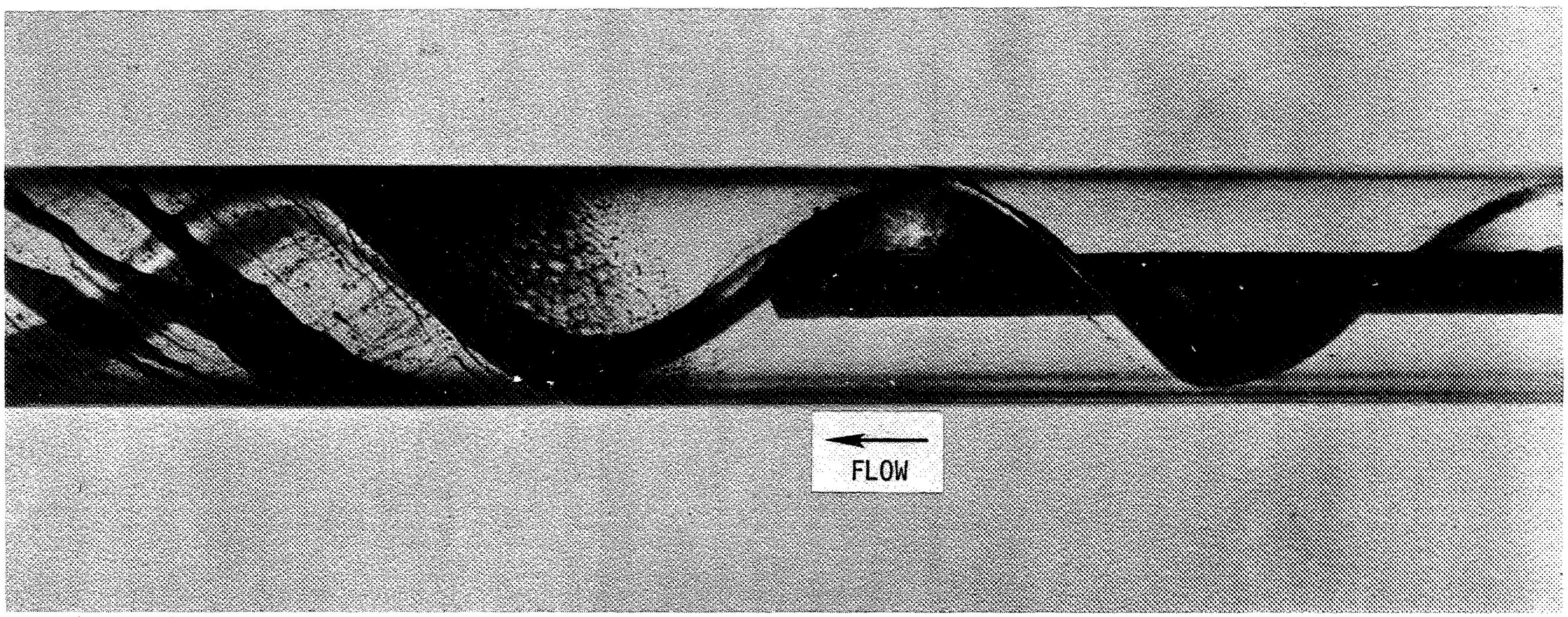

Figure 12. Flow Pattern at End of Swirler Wire Plug Insert (From Reference 5).

In addition to performing chemical analyses on the loop components, the vapor impingement areas of Stage 1, 2, 4 and 6 blades were also analyzed for C, O, H, N. As can be seen in Table IV, no significant change resulted from the 10,000 hours exposure.

Also, analyses were performed on three layers of the 0.002-inch thick Cb-1Zr dimpled foil. Table V summarizes these results and shows that the oxygen increased by a maximum of 293 ppm. It should be noted that this is equivalent to an increase of 6 ppm in the 0.1-inch pipe wall assuming uniform distribution across the wall; and if all the foil contamination is assumed to occur from one side. This calculated increase is in reasonably good agreement with the 14 ppm measured increase shown in Table III.

As is also shown in Table V, the carbon concentration of the foil increased significantly. This increase is currently not understood; however, a significant increase in the carbon concentration was also observed for the thermal insulation foil from the 5000 hour Cb-1Zr test previously reported.[(6)]

c) Weight and dimensional changes

The pre- and posttest weight and dimensional measurements have been performed on the ten turbine simulators nozzles and blades. Neither the weights shown in Table VI nor the nozzle dimensions shown in Table VII showed any significant change due to the 10,000 exposure to potassium vapor.

B. 1900°F Lithium Loop

On May 18, 1970, the 1900°F Lithium Loop completed the initial 2500 hours of testing and was shut down as planned. The fuel element test section was removed from the loop, disassembled, and reloaded with new fuel element specimens. The test section was rewelded into the loop and final preparations are being made to fill the loop and continue testing for an additional 5000 hours.

1. Loop Operation

The 1900°F Lithium Loop successfully completed the planned initial 2500 hours of testing as scheduled. Loop operating temperatures just prior to the shutdown are shown in Figure 13. After installation of

[(6)] Hoffman, E. E. and Holowach, J., Cb-1Zr Rankine System Corrosion Test Loop, Potassium Corrosion Test Loop Development Topical Report No. 7, NASA-CR-1509, 1970 p. 250.

TABLE IV

RESULTS OF CHEMICAL ANALYSIS OF TURBINE SIMULATOR BLADE SPECIMENS*
FOLLOWING THE 10,000-HOUR TEST

Specimen Description	Nozzle Inlet Temperature, °F		Chemical Analysis, ppm O	N	H	C
Stage No. 1 Blade	2142		12	3	< 1	1700
Mo-TZC Alloy			6	6		1800
		Avg.	9	4	< 1	1750
Stage No. 2 Blade	1890		3	3	< 1	1700
Mo-TZC Alloy			8	6		1800
		Avg.	5	4	< 1	1750
Stage No. 10 Blade	1471		14	4	< 1	1600
Mo-TZC Alloy			9	5		1500
		Avg.	11	4	< 1	1550
Before Test			1	1	< 1	1600
			5	1		1600
		Avg.	3	1	< 1	1600
Stage No. 6 Blade	1664		19	9	1	1600
Cb-132M Alloy			17	12	1	1600
		Avg.	18	11	1	1600
Before Test			7	7	< 1	1900
			15	8		1500
		Avg.	11	7	< 1	1700

* Chemical analysis sample taken from the vapor impingement region of each blade.

TABLE V

RESULTS OF CHEMICAL ANALYSIS OF Cb-1Zr FOIL*
TAKEN FROM THE VAPOR CARRYOVER TUBE FOLLOWING THE 10,000-HOUR TEST

Specimen	Estimated Temperature °F		Chemical Analysis, ppm O	N	H	C
Outer Layer	1000		498	37	7	284
			501	32	3	248
		Avg.	499	34	5	266
Middle Layer	1350		332	32	4	150
			348	31	3	168
		Avg.	340	31	3	159
Inner Layer	1800		381	35	2	137
			305	14	7	133
		Avg.	343	24	4	135
Before Test			199	30	2	76
			213	32	2	79
		Avg.	206	31	2	77

*Chemical analysis sample taken from the start of the heat rejection zone. Foil thickness 0.002 inch.

TABLE VI

WEIGHTS OF TURBINE SIMULATOR NOZZLES AND BLADES BEFORE AND AFTER 10,000 HOURS EXPOSURE IN THE T-111 RANKINE SYSTEM CORROSION TEST LOOP

			Weight,[a] grams					
			Blade			Nozzle		
Stage No.	Material	Temp. °F	Before Test	After Test	Change	Before Test	After Test	Change
1	Mo-TZC	2142	25.1496	25.1490	-0.0006	265.4495	(b)	-
2	Mo-TZC	1890	25.3640	25.3640	0.0000	263.9073	263.9007	-0.0066
3	Mo-TZC	1834	25.1849	25.1832	-0.0017	257.1087	257.0958	-0.0129
4	Mo-TZC	1774	25.3275	25.3262	-0.0013	257.6389	257.6286	-0.0103
5	Mo-TZC	1716	25.1817	25.1803	-0.0014	257.6719	257.6620	-0.0099
6	Cb-132M	1664	26.5918	26.5918	0.0000	272.0057	272.0074	+0.0017
7	Mo-TZC	1611	25.3579	25.3565	-0.0014	256.7299	256.7234	-0.0065
8	Mo-TZC	1562	25.2678	25.2656	-0.0022	253.1244	253.1165	-0.0079
9	Cb-132M	1514	26.5369	26.5364	-0.0005	268.8649	268.8658	+0.0009
10	Mo-TZC	1471	25.2144	25.2127	-0.0017	251.5495	251.5372	-0.0123

[a] Average of duplicate measurements

[b] Not measured; bonded to T-111 alloy fitting

TABLE VII

TURBINE SIMULATOR NOZZLE THROAT DIAMETERS BEFORE AND AFTER 10,000 HOURS EXPOSURE IN THE T-111 RANKINE SYSTEM CORROSION TEST LOOP

Stage No.	Material	Temp. °F	Diameter,[a] inches Before Test	After Test	Change
1	Mo-TZC	2142	0.089175	0.089327	+ 0.000152
2	Mo-TZC	1890	0.087925	0.087810	- 0.000115
3	Mo-TZC	1834	0.096380	0.096215	- 0.000165
4	Mo-TZC	1774	0.108275	0.108307	+ 0.000032
5	Mo-TZC	1716	0.118060	0.118050	- 0.000010
6	Cb-132M	1664	0.129225	0.129185	- 0.000040
7	Mo-TZC	1611	0.145737	0.145800	+ 0.000063
8	Mo-TZC	1562	0.159837	0.159790	- 0.000047
9	Cb-132M	1514	0.178425	0.178562	+ 0.000137
10	Mo-TZC	1471	0.198644	0.198595	- 0.000049

[a] Measurements performed by Sheffield Corporation, Dayton, Ohio. Reported accuracy is 0.00005 inch and is the average in duplicate measurements 90° apart.

Date 5-18-70 Test Time 2500 Hrs.
Chamber Pressure 2.3×10^{-9} Torr
Heater 39 A 305 V
Flow 2.2 mv 1.0 GPM

TEMPERATURES IN °F

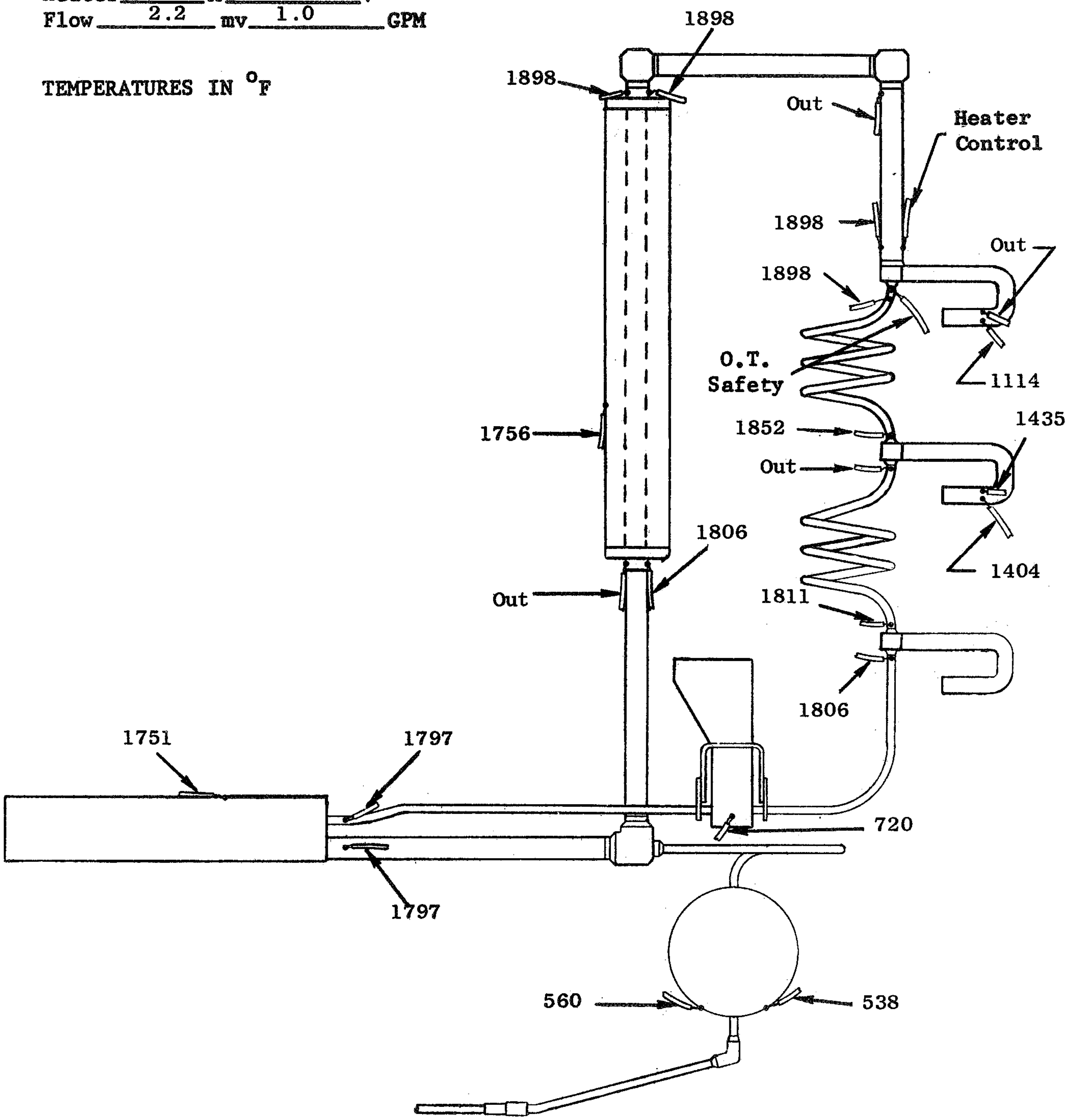

Figure 13. 1900°F Lithium Loop Operating Temperatures - 2500 Hours.

the constant voltage transformer[7] the loop operated very well, and no more shutdowns due to voltage instabilities were encountered. Except for the first 100 hours, the loop operated for the entire test period on automatic control with no manual adjustments necessary.

2. Test Chamber Environment - Partial Pressure Gas Analysis

The chamber pressure and partial pressure of the various gaseous species in the test chamber during the entire 2500 hours are shown in Figure 14. During the last half of the test there is a general trend for the partial pressures to level off. Although data points are shown at 250-hour intervals for clarity, the chamber pressure is monitored continuously, and partial pressure gas analyses are performed at least once every 24 hours (except for weekends when no surveillance is used).

3. Loop Shutdown

Upon completion of the 2500 hours of testing, the lithium was hot dumped (1000°F) into the surge tank and then immediately drained into the alkali metal transfer system charge pot and a sample taken. The loop was then pressurized through the blowdown line to further remove any lithium. Analysis of the lithium compared to the pretest analysis is shown in Table VIII. The increased oxygen is consistent with dissolution of oxygen from the T-111 loop components and has been observed previously.[7] The increased nitrogen is somewhat unusual; the only known significant sources of nitrogen are the clad UN fuel pellets and the ASTAR 811CN corrosion specimens.

Complete explanation, therefore, of the increased nitrogen must await the final posttest evaluation of the clad fuel element specimens and the ASTAR 811CN specimens.

4. Lithium Distillation

After cooling all components to room temperature, the vacuum chamber bell jar was removed. Visual inspection of the loop showed all components to be in excellent condition. The Cb-1Zr dimpled foil thermal insulation was removed from the fuel specimen test section. The loop with foil removed is shown in Figure 15. Two new layers of foil were applied and

(7) Advanced Refractory Alloy Corrosion Loop Program Quarterly Progress Report No. 20, NASA-CR- 72739, GESP- 491, May 11, 1970.

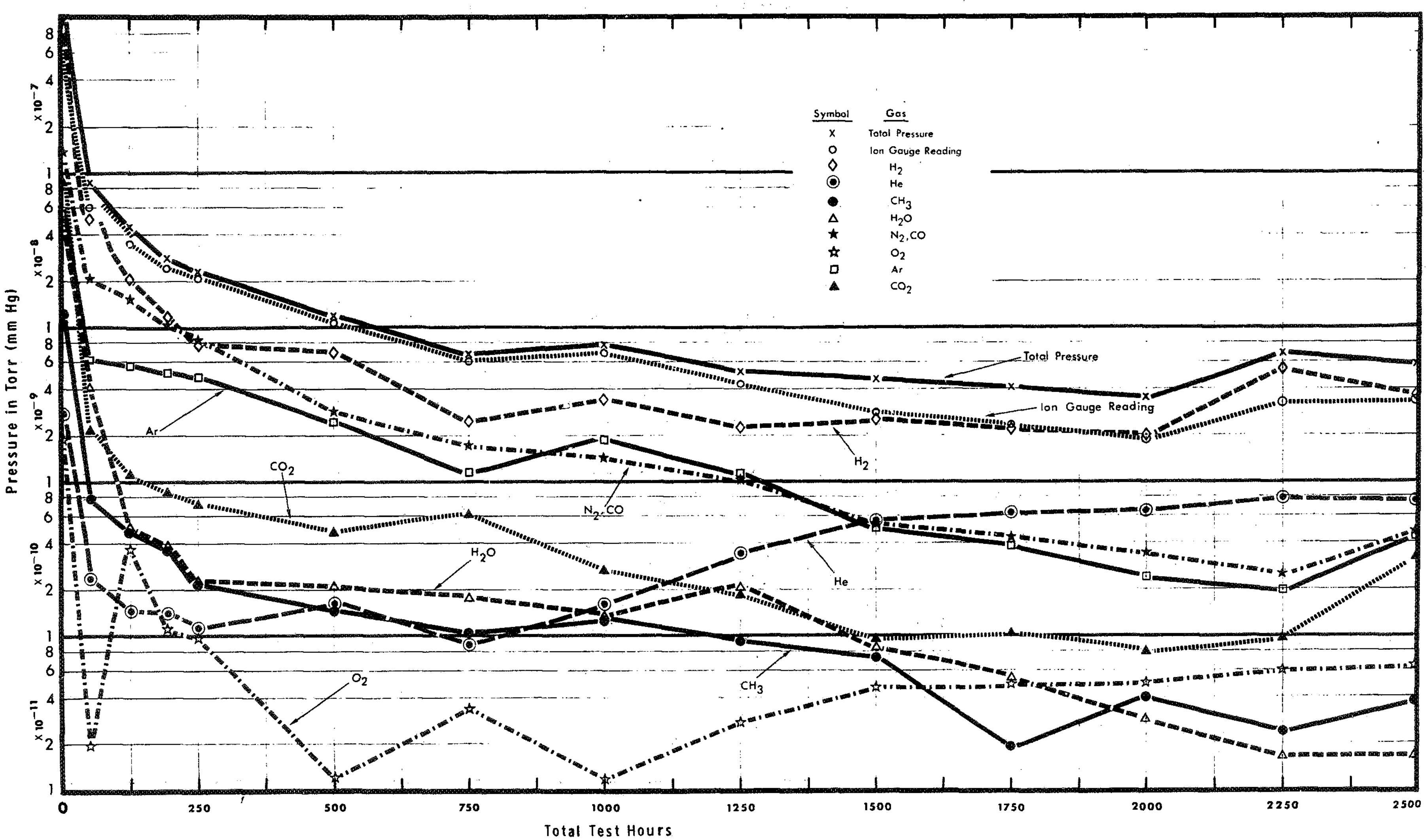

Figure 14. Test Chamber Environment During Testing 1900°F Lithium Loop.

TABLE VIII

LITHIUM ANALYSIS - 1900°F LITHIUM LOOP

	Pretest	Posttest 2500 Hrs
Oxygen	28	149
Nitrogen	2, 3	118
Carbon	76	99
Silver	< 5	< 5
Aluminum	5	25
Boron	< 75	< 75
Barium	< 50	< 75
Beryllium	< 5	< 5
Calcium	25	5
Columbium	< 25	< 25
Cobalt	< 5	< 5
Chromium	< 5	< 5
Copper	5	< 5
Iron	5	< 5
Magnesium	5	< 5
Manganese	< 5	< 5
Molybdenum	< 5	< 5
Sodium	--	< 75
Nickel	< 5	< 5
Lead	< 50	< 50
Silicon	< 5	5
Tin	< 25	< 25
Strontium	50	50
Titanium	< 25	< 25
Vanadium	< 25	< 25
Zirconium	< 25	< 25

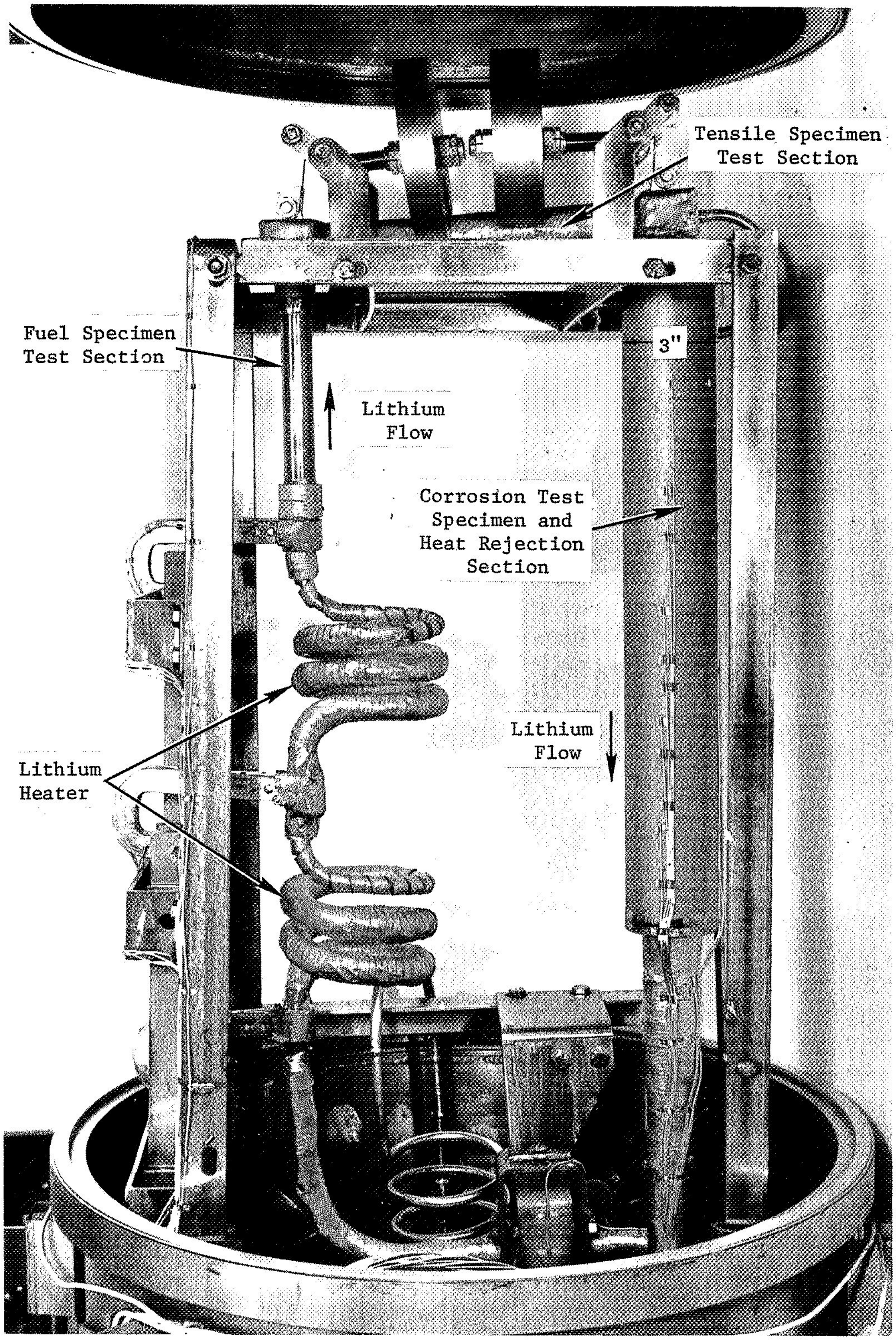

Figure 15. 1900^{o}F Lithium Loop Following Initial 2500 Hours of Operation. Thermal Insulation Foil Removed from Fuel Specimen Test Section. (Orig. P70-5-19D)

a tungsten resistance furnace placed around the fuel specimen test section (as shown in Figure 16); finally the bell jar was replaced on the spool piece. After evacuating the loop and vacuum chamber, the bakeout ovens and the surge tank heater were turned on to bring all loop temperatures to a minimum of 400°F. Residual lithium was then distilled from the fuel test section for eight hours at 1900°F utilizing the tungsten resistance heater. During distillation, the loop was evacuated using the getter-ion pump in the lithium transfer system. After cooling all components to room temperature, the bell jar and distillation furnace were removed and all electrical, gas, and liquid metal lines were disconnected from the spool piece.

5. Removal and Replacement of Fuel Element Test Section

After completion of the above operations, the loop was moved from the test area to the weld laboratory. The loop and spool pieces were then mounted in the motor driven rotary weld fixture which was installed in the eight-foot-diameter weld chamber as shown in Figure 17. The weld chamber was then evacuated and backfilled with helium according to GE-NSP Specification 03-0025-00-A. The fuel test section was removed by cutting the loop with a tubing cutter at locations (1) and (2) shown in Figure 18; and the open ends of the loop plugged with expandable stoppers prior to opening the weld chamber to air. The fuel specimen subassembly was radiographed to verify the integrity of the specimens, and finally a cut was made at location (3) shown in Figure 18 to remove the fuel specimens. Visual examination showed all components to be in excellent condition, and no traces of lithium were present, indicating the effectiveness of the 1900°F distillation. The appearance of the T-111 clad fuel specimens and Mo-TZM spacers before and after the 2500 hours lithium exposure is shown in Figure 19. The posttest specimens are shown in the as-distilled condition and were not further cleaned prior to the photograph. Some discoloration of the Mo-TZM spacers was observed. After removal of the fuel specimens from the housing, they were radiographed to verify their integrity; no difference could be seen between the pretest and posttest radiographs.

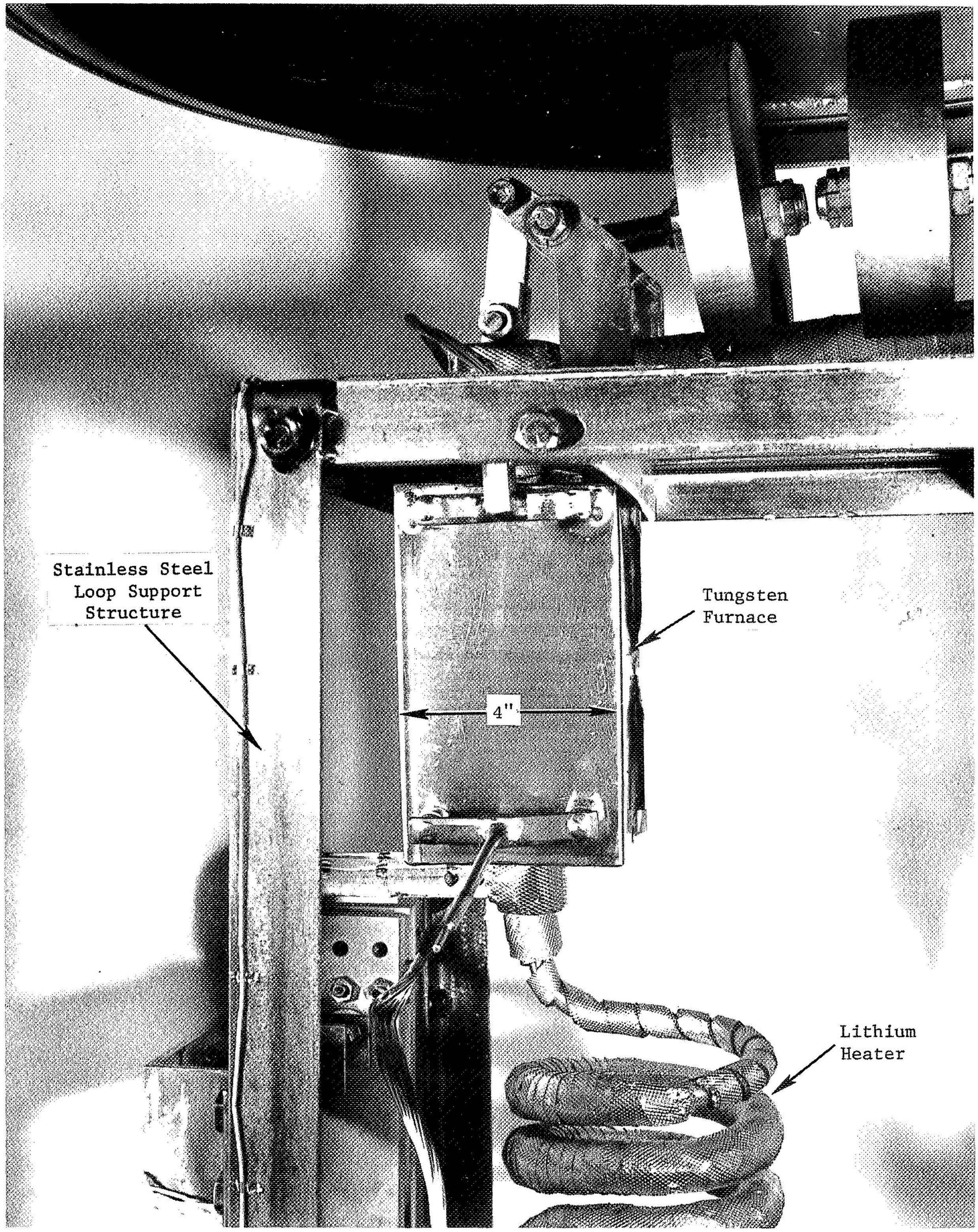

Figure 16. 1900°F Lithium Loop Showing Tungsten Furnace in Position Around Fuel Specimen Test Section as it was Used for Lithium Distillation and Postweld Annealing. (Orig. P70-5-19E)

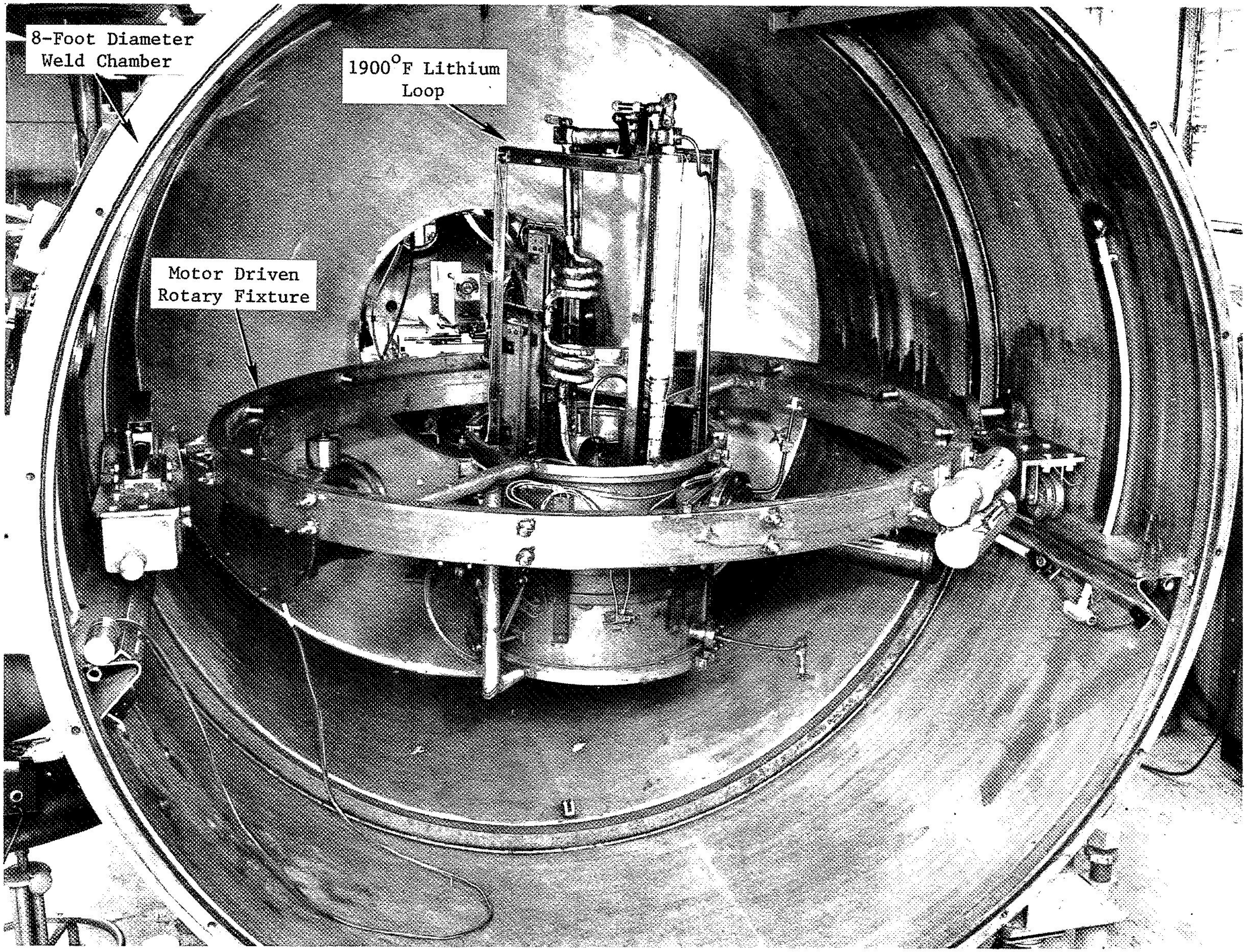

Figure 17. 1900°F Lithium Loop Mounted on Motor Driven Rotary Fixture in 8-Foot Diameter Weld Chamber. (Orig. P70-6-3A)

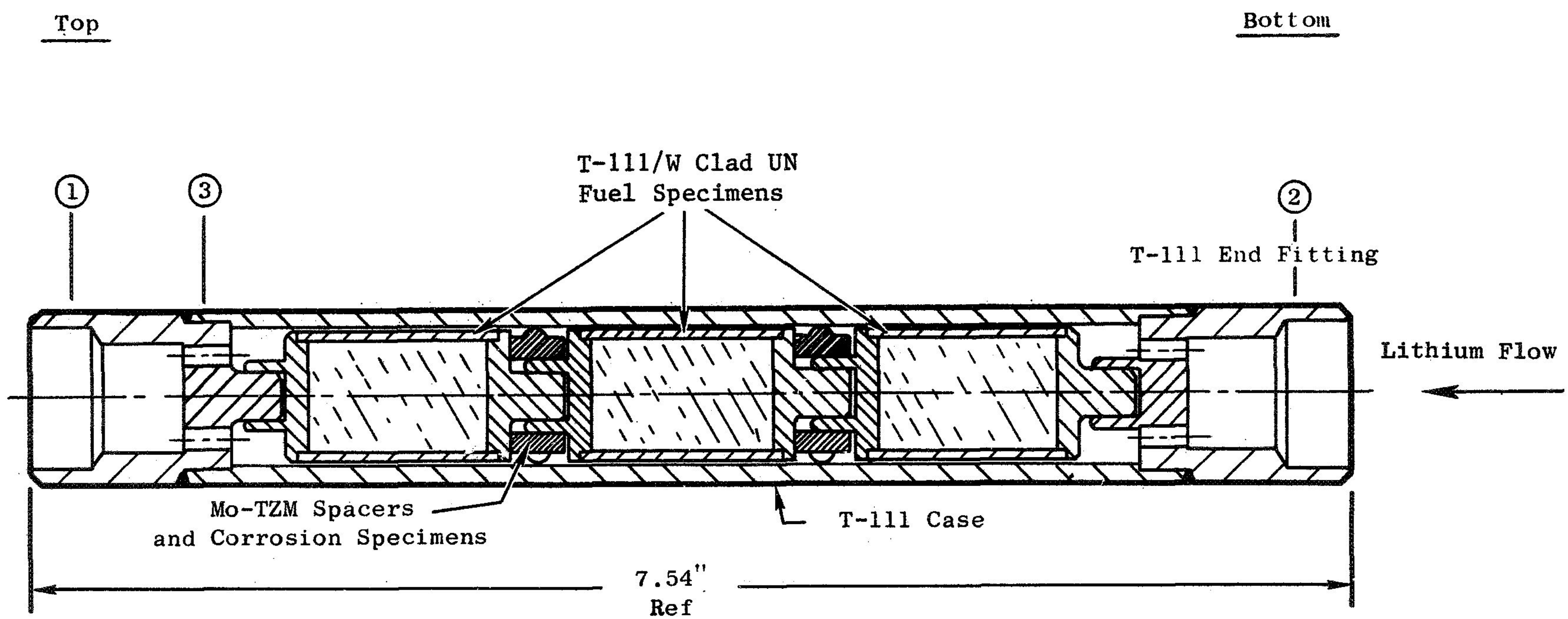

Figure 18. Fuel Specimen Test Section - 1900°F Lithium Loop.

(470-1)

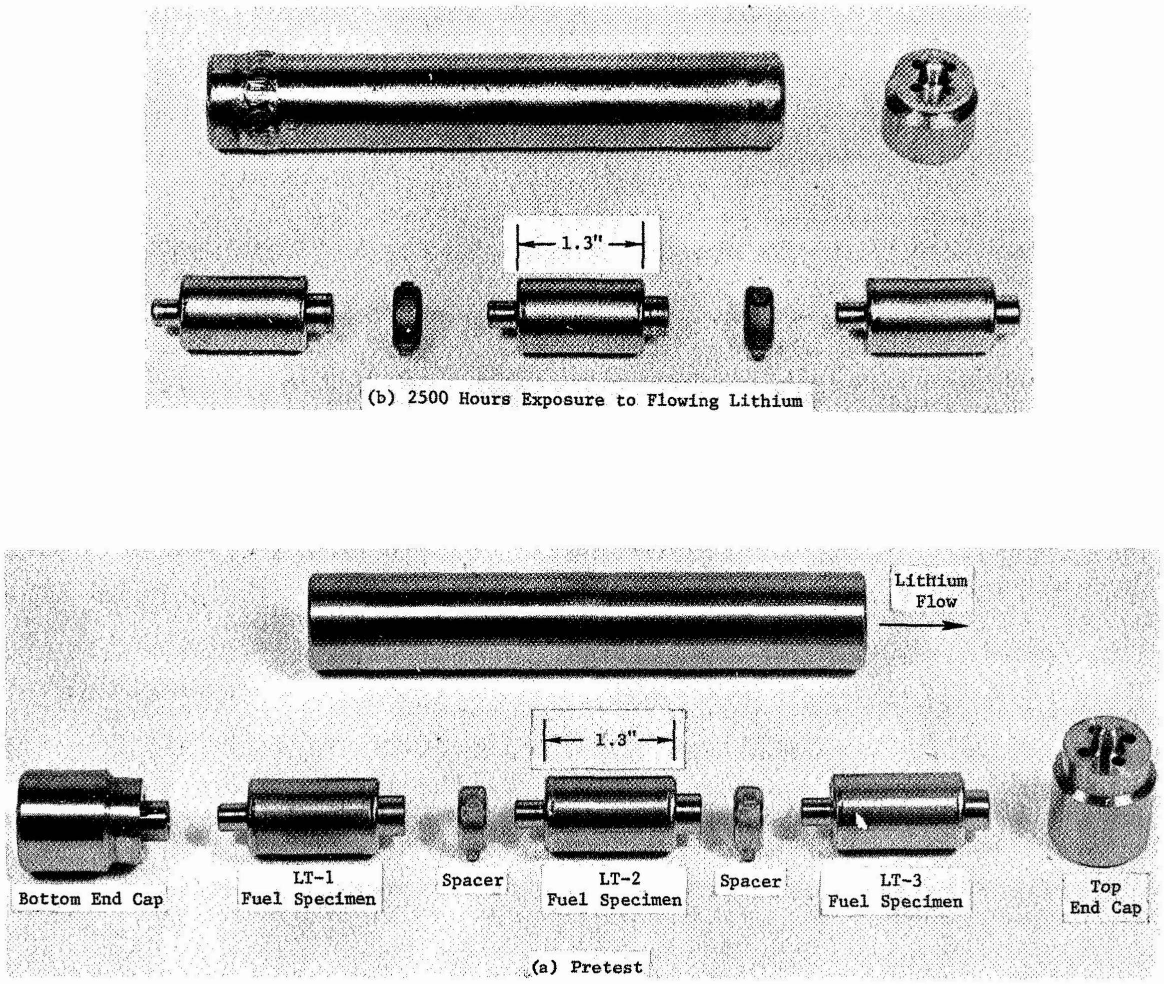

Figure 19. Components of Clad Fuel Element Specimen Subassembly (a) Before and (b) After 2500 Hours Exposure to Flowing Lithium at 1900°F. All Components are T-111 Except the Spacers Which are Mo-TZM. (Orig. P70-6-8A, P69-9-31C)

After pickling, the fuel test section was reassembled reusing the same T-111 end caps and housing. Specimens were inserted in the following sequence:

Lithium Flow ↓

1. T-111 clad fuel element LT-5: untested.
2. Mo-TZM spacer: untested.
3. T-111 clad fuel element LT-2: tested during the initial 2500 hours of the 1900°F Lithium Loop.
4. Mo-TZM spacer: tested during the initial 2500 hours of the 1900°F Lithium Loop.
5. T-111 clad fuel element LT-6: untested containing a longitudinal clad defect (planned) as shown in Figure 20.

This subassembly was then rewelded at location (3) (Figure 18) and then welded back into the loop at locations (1) and (2), according to GE-NSP Specification 03-0025-00-A. The loop was then removed from the weld chamber and fixture, and subsequently moved back to the test area. All thermocouples were repaired; two layers of Cb-1Zr dimpled foil were applied to the fuel test section; and the tungsten resistance heater was reassembled around the fuel test section. The spool piece and bell jar were placed on the vacuum chamber sump, and pumpdown of the chamber was initiated in preparation for the postweld anneal. The anneal was performed for one hour at 2400°F with a maximum chamber pressure of 6×10^{-6} torr. During the anneal all vacuum chamber bakeout heaters and the surge tank heater were turned on to maintain loop temperatures at a minimum of 400°F. Also, during the anneal the loop was evacuated through the blowdown line to prevent pressure buildup by the high temperature. The loop was leak checked before and after the anneal by pressurizing the loop with 50 psia argon through the blowdown line while monitoring the vacuum chamber for argon with the partial pressure gas analyzer. No leaks were detected during either check.

After removal of the annealing furnace, the fuel specimen test section will be wrapped with Cb-1Zr dimpled foil and other final preparations made prior to filling the loop with lithium and returning to the 1900°F test conditions.*

*On July 31, 1970 the loop was brought to test conditions.

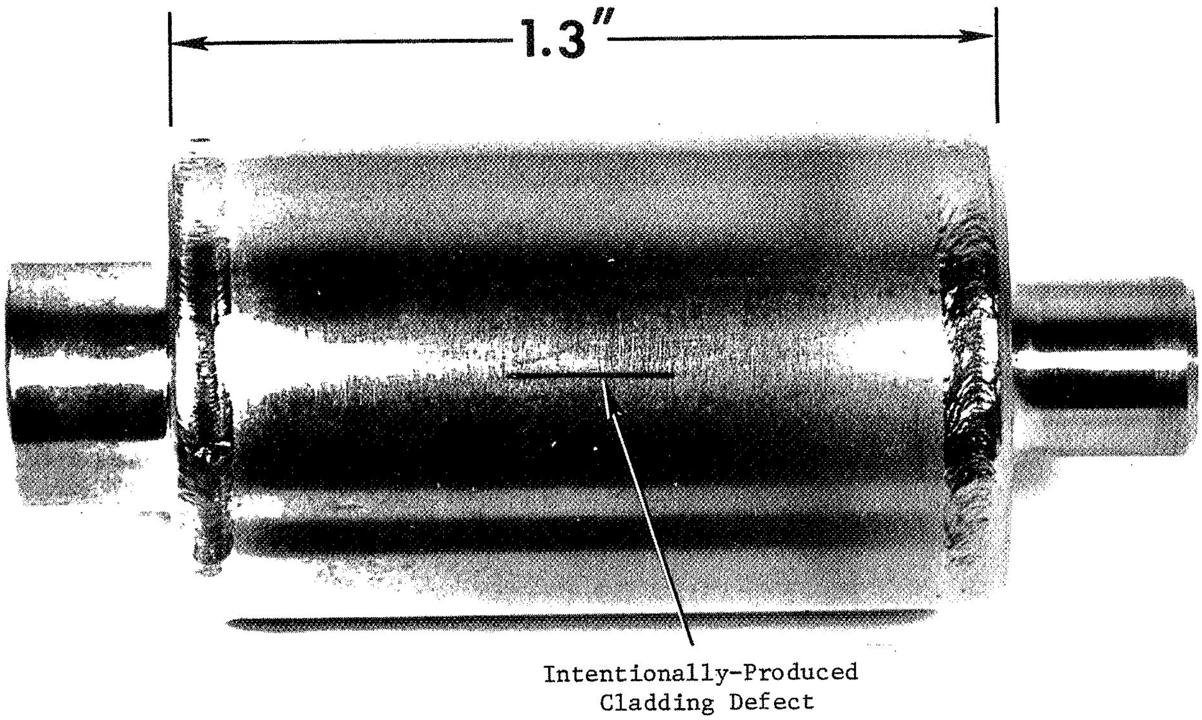

Figure 20. T-111 Clad Fuel Specimens (LT-6) Illustrating 1/4-Inch Long Clad Defect Prior to Testing in the 1900°F Lithium Loop. (P70-6-1A)

6. Posttest (2500 hours) Evaluation

The weight change of the T-111 clad fuel element specimens and Mo-TZM spacers was measured at the completion of the 2500 hours of testing. The results, summarized in Table IX. show a 2-3-mg weight loss on the fuel specimens and a 3-4-mg weight gain on the spacers. The relatively high percent change in weight of the spacers correlates with the observed discoloration shown in Figure 19. Metallographic inspection will be performed on the spacers at GE-NSP. All posttest evaluation of the fuel elements will be performed at NASA-Lewis Research Center.

TABLE IX

WEIGHTS OF FUEL ELEMENTS AND SPACERS BEFORE AND AFTER 2500-HOUR EXPOSURE TO FLOWING LITHIUM AT 1900°F

Fuel Specimen No.[(a)]	Before Test	After Test	Change
LT-1	135.9875 g	135.9853 g	-0.0022 g
LT-2	135.2553 g	135.2524 g	-0.0029 g
LT-3	135.5796 g	135.5769 g	-0.0025 g
Spacer Number[(b)]			
LT-1A	8.7253 g	8.7289 g	+0.0036 g
LT-2A	8.6406 g	8.6446 g	+0.0040 g

(a) T-111 clad UN.

(b) Mo-TZM.

IV. FUTURE PLANS

Continue posttest evaluation of loop components from the T-111 Rankine System Corrosion Test Loop.

Initiate remaining planned 5000 hours of testing on the 1900°F Lithium Loop.

Complete topical report on potassium reflux capsule tests and lithium thermal convection capsule tests on advanced tantalum alloys.

PREVIOUSLY PUBLISHED PROGRESS REPORTS FOR THIS CONTRACT

Quarterly Progress	For Quarter Ending
Report No. 1 (NASA-CR-54477)	July 15, 1965
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